

**SEDIMENT ASSESSMENT:  
A GUIDE TO STUDY  
DESIGN, SAMPLING AND  
LABORATORY ANALYSIS**

**JULY 1993**



**Ministry of  
Environment  
and Energy**



ISBN 0-7778-1511-7

**SEDIMENT ASSESSMENT: A GUIDE TO  
STUDY DESIGN, SAMPLING AND LABORATORY ANALYSIS**

JULY 1993



Cette publication technique  
n'est disponible qu'en anglais.

Copyright: Queen's Printer for Ontario, 1993  
This publication may be reproduced for non-commercial purposes  
with appropriate attribution.

PIBS 2595E



**SEDIMENT ASSISSMENT: A GUIDE TO  
STUDY DESIGN, SAMPLING AND LABORATORY ANALYSIS**

Report prepared by:

R. Jaagumagi and D. Persaud  
Water Resources Branch

JULY 1993



### Acknowledgement

The authors are indebted to Dave Hollinger, John Parks and Arnie Laine of Northwestern Region and to Dr. Peter Kauss of the Great Lakes Section for their invaluable comments and suggestions.

Certain sections of this report have appeared earlier as part of the MOEE series "Evaluating Construction Activities Impacting on Water Resources: Handbook for Dredging and Dredged Material in Ontario".



## Table of Contents

Introduction .....	1
Section I. Design of Sediment Sampling Programs .....	2
1.1 General Considerations .....	2
1.1.1 Defining the Objectives of a Sediment Study .....	2
1.1.2 Designing a Sediment Survey .....	3
Section II Development of Sediment Assessment Studies for Specific Types of Projects .....	10
2.1 Lakefilling .....	10
2.1.1 Design Considerations .....	10
2.1.2 Survey Design .....	10
2.2 Dredge Disposal .....	11
2.2.1 Design Considerations .....	11
2.2.2 Summary of Sampling Program Design for Dredging Projects .....	12
2.3 Sediment Assessment Studies/ Surveillance and Monitoring .....	13
2.3.1 Design Considerations .....	13
2.3.2 Survey Design .....	13
2.3.3 Summary of Sampling Program Design for Surveillance and Monitoring Studies .....	14
2.5 Spills Assessment and Clean-up .....	15
Section III. Biological Assessment of Sediment Contamination .....	15
3.1 Benthic Surveys .....	15
3.1.1 Community Structure/ Functional Analysis .....	16
3.1.2 Biota Tissue Residue Analysis .....	19
3.2 Fish .....	20
3.2.1 Tissue Residue Analysis .....	20
3.3 Sediment Bioassays .....	20
Section IV. Sediment Sampling for Chemical and Biological Assessment .....	21
4.1 Sampling Devices .....	21
4.2 Field Records .....	23
4.2.1 Positioning .....	23
4.2.2 Field Notes .....	23
4.2.3 Physical Analysis .....	23
4.3 Field Storage and Handling .....	24
4.3.1 Grab Samples .....	24
4.3.2 Cores .....	24
4.3.3 Field Storage and Handling for Samples for Partial Geochemical Leaching .....	24
4.3.4 Laboratory Handling and Storage .....	25
4.3.5 Archive and Duplicate Samples .....	25
4.3.6 Sample Splitting Report .....	25
Section V. Sample Analysis .....	25
5.1 Particle Size Analysis .....	25
5.2 Trace Inorganic Chemical Analysis .....	25
5.2.1 Nutrients .....	25
5.2.2 Trace Metal ("Bulk Metal"). .....	26
5.2.3 Trace Metal (Partial Leaching). .....	26
5.2.4 Other Inorganic Compounds. ....	26

## Table of Contents (cont'd)

5.3 Trace Organic Chemical Analysis . . . . .	27
5.3.1 PCB and Organochlorine Pesticides . . . . .	27
5.3.2 Polyaromatic Hydrocarbons (PAHs) . . . . .	27
5.3.3 Other Compounds . . . . .	27
5.4 Miscellaneous Analysis . . . . .	27
5.4.1 Organic/Inorganic Carbon. . . . .	27
5.4.2 Solvent Extractables (Oil and Grease) . . . . .	28
Section VI. Quality Assurance/Quality Control Considerations . . . . .	28
6.1 Introduction . . . . .	28
6.2 Elements of QA/QC Program . . . . .	29
6.3 Implementation of QA/QC Program . . . . .	29
6.3.1 Sampling . . . . .	29
6.3.2 Sample Handling and Processing . . . . .	30
6.3.3 Laboratory Analysis . . . . .	30
Section VII. Data Interpretation . . . . .	31
Section VIII. References . . . . .	33
Appendix 1 Sources of Historic Information	
Appendix 2 Operational Evaluation of Sampling Devices	

## FOREWORD

The information in this document has been compiled to complement the Provincial Sediment Quality Guidelines and the Fill Quality Guidelines for Lakefilling in Ontario by providing the basis for uniformity in sediment information gathering procedures. Although various sampling protocols have been described in the literature as part of individual studies, there is generally no uniform method that would facilitate ease of interpretation and comparison of sediment results.

In order for a sediment survey to be of maximum benefit, it must meet all of the stated objectives of the study. This requires that an adequate preliminary plan be prepared. This document offers suggestions on survey designs, and, while not advocating that a particular methodology or protocol be followed, it is hoped that by avoiding common pitfalls in sampling design and implementation, many of the limitations of such data collections can be overcome.

Details on sampling devices and design strategies are included in the appendices. Details on laboratory procedures are not included, instead the appropriate sources for such information are referenced.

## INTRODUCTION

The Provincial Sediment Quality Guidelines (PSQG) (Persaud *et al.* 1992) provide a means of assessing the biological significance of contaminants in sediments. The PSQGs have identified a number of sediment-related activities to which the guidelines would be applied, such as lakefilling, dredging, and sediment monitoring studies. This document complements the PSQG document by outlining several considerations for sediment studies, such as appropriate study design, choice of sampling devices, sample analysis and quality assurance/quality control (QA/QC) measures.

The sediment guidelines also contain provisions for management decisions to be made both on the basis of the chemical quality of the sediments and on the basis of biological effects. Therefore, exceedances of the various guideline levels provide a starting point for additional assessment of possible biological effects.

The PSQGs have identified a number of situations where additional sediment/biological studies may be necessary. Principally, where sediment contaminant levels exceed the Severe Effect Level or a combination of the Lowest Effect Level and local background, additional sediment/ biological work will have to be considered. In such cases, a clear definition of the potential biological effects is necessary before management decisions can be

made.

The assessment of biological effects is a complex task. No single study component is capable of providing the information necessary to make informed decisions on the management of contaminated sediments. In addition to delineating the geographical extent and ecological severity of contamination, biological studies are necessary to determine toxic and/or bioaccumulant effects on organisms. Sediments are also recognized as a potential source of contaminants to the water column, and to water column organisms. Since both aspects are recognized as major factors that influence management decision/action, this information is critical to sediment assessment. Therefore, where the Severe Effect Level of the PSQGs or a combination of the Lowest Effect level and local background have been exceeded, the development of a sampling program for sediments must of necessity include biological sampling of sediment dwelling organisms, their predators, the water column, and effects on those organisms, such as fish, that are resident in the water column.

Sediment assessment is accomplished through the collection and analysis of samples for physical and chemical characterization of bottom sediments. Biological assessment is accomplished through: benthic community assessment, which considers the effects of contaminants on the composition of the benthic community (i.e., types and numbers of organisms); tissue residue analysis which determines the availability of the contaminants to sediment

dwelling organisms; and sediment bioassays, which measure the potential toxicity of the contaminants to benthic organisms. Effects on the water column are estimated through sediment bioassays that use water column organisms, and through biomonitoring (i.e., direct field testing) with organisms such as mussels.

The information in this document provides the basis for a uniform approach to sampling and chemical analysis, which is necessary for the proper comparison of results to the PSQGs. This document discusses sampling undertaken to characterize chemical quality and sampling for biological information on benthic organisms.

Proponents of sediment related studies should consider the information presented in this document as a preliminary guide. The single most important source of information available to proponents is the local MOEE Regional or District Office. It is strongly advised that all proponents discuss any proposed undertaking with Regional staff prior to commencing work. Not only does this make available to the proponent a wealth of specific information, it also ensures that any specific concerns relating to the study area will be addressed.

## SECTION I. DESIGN OF SEDIMENT SAMPLING PROGRAMS

### GENERAL CONSIDERATIONS

A basic requirement for an effective sampling program is a well devised study plan that clearly outlines the objectives of the study and lays out the appropriate procedures for obtaining the information. Most sediment studies are conducted to assess the chemical quality of the sediment, usually through comparison of the results with guidelines established for sediment evaluation. These are often undertaken in support of sediment studies to: establish baseline data; provide data for State of the Environment reporting; investigate fate and effects of contaminants; and to monitor impacts of discharges.

Complementary to chemical evaluation studies are biological studies which are carried out concurrent with, or subsequent to, a chemical evaluation.

Biological studies are used to determine possible effects of contaminants in sediment on resident organisms. These can range from toxic effects on individuals to long-term sub-lethal effects at the community level.

There are four broad categories of projects normally requiring sediment assessment: dredging, lakefilling, spills clean-up, and surveillance and monitoring programs. The degree of detail required for each is different and also varies from area to area within individual categories.

The amount of information that must be gathered will depend on how much background data is available for the site. A review of historic data can help to focus information-gathering studies by identifying concerns, as well as highlighting gaps in information. In the absence of historic data, a more intensive effort may be required to obtain adequate decision-making information.

The design of a sediment sampling program can benefit significantly from additional input from staff in the MOEE Regions. It is recommended that development of a study plan proceed in consultation with MOEE Regions.

### 1.1 Defining the Objectives of a Sediment Study

The reasons for undertaking the study and the questions to be answered form the basis of the study design. Therefore objectives need to be clearly defined and described in detail before any survey plans are developed. When the objectives of the study are clearly laid out, they ensure that the essential work is carried out (according to budget allocations and schedule) and that non-essential activities are discarded.

Where the aim of the study is broad in scope, such as a complete environmental assessment of an area, the study should be divided into specific sub-objectives. There can be a number of these specific aims, such as comparison of concentrations among sites, mapping of contaminant distributions, and/or determination of biological effects. The study can then be divided into separate components that are designed to answer the specific questions. Ensuring that the specific concerns are addressed and that

effort is not expended on those components that do not directly address the concerns is usually the most critical part of a sediment assessment study.

Identification of the specific aims of the study enables the proponent to select the most suitable tests and analyses and the best strategy for obtaining the samples. These would include both chemical and biological tests such as:

- bulk sediment analysis (e.g., trace metals, persistent organic compounds such as PCBs, organochlorine pesticides, PAHs, chlorophenols, etc)
- sediment geochemical fractionation (metals)
- bioaccumulation or tissue residue analysis (invertebrates, fish, plants, etc)
- benthic invertebrate community structure analysis
- sediment bioassays

In most cases, the study aims will be specific to the area and the type of project the study is intended to support. The questions to be answered will determine the scope of the study. For example, if the aim is to assess the impacts of a specific sediment "hotspot" or a point source discharge, the design of the sampling program will be different from a study where the aim is a general assessment of the "health" of a waterbody. Ultimately, the success of the project will depend on how precisely the aims can be defined and how well the strategy to acquire the necessary information is laid out. In most cases where a sediment clean-up action may be required, or where dredging has been proposed, a detailed characterization of the area is in the best interests of the sponsoring agency/industry. In general, a clear delineation of the boundaries of a contaminated area could mean a significant reduction in the removal of excess (uncontaminated) material with corresponding cost savings in material removal (dredging) and disposal.

### 1. 1. 2 Designing a Sediment Survey

The purpose of the study design is to derive the best strategy for obtaining relevant information in

the most cost effective manner (from both a financial and human resource perspective).

The development of a sediment sampling program would typically proceed through a number of steps.

#### i. *Review existing data*

The initial step in designing a sampling program is to undertake a careful review of historical data. The purpose of this review is to characterize the existing sediments in terms of sediment type and contaminant concentrations, and to identify any gaps in the data. The proponent should be aware that the historical data may be incomplete, especially in the number of parameters measured, or out of date if the information is more than a couple of years old.

The PSQG document has laid out a mandatory parameter list for sediment analysis which represents the minimum number of parameters to be included in the analysis. However, in certain cases, additional parameters may also be necessary, either to achieve the objectives of the study or as a requirement by MOEE.

The data review should seek to identify all existing and historical contaminant sources to the area, such as industrial/municipal outfalls and urban runoff sources, in order to identify additional potential contaminants for which chemical analysis may be necessary.

Having collected all available data, the proponent should collate the information in order to identify any gaps. The filling of these gaps will assist in defining the sampling program to be undertaken.

One important aspect which is often overlooked when developing a sediment assessment program is an inspection of the site to ensure that the historical information is still accurate. The site inspection should include identification of new sources of contaminant input (industries, outfalls); discussion with municipal planning agencies, and; discussion with district fish and wildlife agencies and MOEE Regional and District Offices. The site inspection will enable the proponent to obtain a better under-

standing of public perceptions and concerns, and identify any difficulties which could hinder the sampling operation.

Where historical information is not available, it is advisable to undertake a preliminary survey to assist in the planning of the detailed program.

At the conclusion of this stage there should be sufficient information to plan a sampling program which will generate the required information with minimal effort and time.

## ii. *Define study area*

a) The study area should extend far enough spatially that it encompasses the entire zone of impact. A primary consideration is the need to delineate a study area large enough such that effects due to the source under investigation can be detected and that the severity of the effect can be determined relative to adjacent, unimpacted areas.

- historic or preliminary data should be used in delineating the study area and focusing the investigation.
- where previous information is not available, a preliminary study should be considered in order to spatially define the study area.
- for investigations that are broad in scope, such as a sediment assessment for State of the Environment reporting or lake basin studies, the aims or terms of reference of the study will define the extent of the study area. The scope and spatial extent will be governed by the case-specific aspects of the project.
- in lentic (standing) water (e.g., lakes), consideration should be given to wave action and current movements in order to project movement and dispersal of discharge from the source under investigation and thus help define the size of the study area.
- in lotic (flowing) water (e.g., rivers), con-

sideration should be given to the flow dynamics of the watercourse and the nature of any discharges (i.e. do materials settle rapidly, or are they carried over long distances) when defining the study area. Preliminary studies or historical data can be of significant value, especially in identifying sources and extent of impact.

b) Determine the duration of the study.

Sediment studies in support of specific concerns, such as those in support of a lakefilling or dredging project (and others designed simply for comparison with the PSQGs), are not intended as long term assessments (i.e., designed to determine changes over an extended period of time). Studies addressing specific concerns are designed to provide information on the existing quality of sediments for immediate use in management decisions. The opposite is usually true for routine sediment assessment (surveillance) or monitoring studies, which are normally designed as long-term studies, with repeated sampling over specified time intervals.

In designing long-term studies that measure seasonal or temporal changes, consideration should be given to the following:

- the location of stations and the number of sampling sites must be chosen with care to ensure that they can be located during future studies, and thus enhance the continuity and comparability of the data (station locations should be adequately documented, e.g., latitude and longitude).
- it is often preferable to sample intensively during the initial survey and then eliminate stations in future surveys, rather than add locations later, when the shortcomings of the initial survey become apparent.
- for some types of assessments, such as those for lakefilling projects, seasonal studies may be necessary in order to determine effects under different wave or current/discharge regimes.

iii. *Determine most suitable study design.*

a) **Sampling Strategies.**

A number of strategies are available for designing sediment sampling programs. The choice of sampling strategy depends on the nature of the problem and the type of area being investigated. Baudo (1990) defines three primary sampling strategies that can be used to develop a suitable design:

1. **Deterministic.**

This design is most often used where previous information is available. Under this approach, stations are located in relation to the specific concerns driving the investigation. The number of sampling stations is determined by how much detail is required on the site in order to address the concerns. Thus, for investigation of a near-shore discharge, for example, stations would be located on the basis of previous knowledge of sediment contaminant distributions or knowledge of plume movements from the discharge. For a dredging project, it may be necessary to segregate areas according to the degree of contamination in order to recommend different management options or disposal modes for the material.

2. **Stochastic.**

The stochastic system is most effective where data will be used for statistical analysis. The station locations are chosen by subdividing the area into equal segments and randomly selecting segments to be sampled (Baudo 1990). This method can be applied to any type of study area, provided the size of the segments is appropriate to the study aims.

3. **Regular Grid System**

Using this system the study area is divided into regularly spaced grids and the sample locations are selected either randomly (see 2. above) or deterministically (see 1. above) from the available grids. This is often the preferred method where little or no previous information is available or where a number of

sources of contaminants may exist. In particular, this method is commonly used where a map of sediment contamination is the desired product, since it provides uniform coverage of the study area.

For most assessment studies, some type of deterministic sampling method is used for the selection of sampling locations, either based on random selection or a regular grid system. Stratification of station locations, based on physical similarity of the sediments (determined through a preliminary survey), is often a necessary modification in random sampling designs, in order to achieve statistical comparability of results among stations. Since stratified random sampling is based on estimates of sampling error, this method requires prior information on the study area, either from previous sampling or from the literature, and can only be used where such information is available. Where review of the aims of the study has identified this method as the optimum survey design, a preliminary survey may be necessary before the final sampling program can be established.

In lentic (standing) water where the investigation is directed towards a specific point-source discharge, a deterministic sampling design is the most common. Using this design, sampling stations are located in a grid or radiating pattern around the source.

- the grid can be a regular grid based on lines intersecting at right angles, with sampling points located at the intersections. The spacing of the lines should be such that it provides adequate coverage of the area, with emphasis on areas of sediment accumulation and other known features.
- a radiating pattern can be used, with "spoke" lines radiating out from the source and intersecting arcs at right angles to these lines. The sampling locations are usually positioned at the intersections of these lines. The spacing of the lines should be based on existing knowledge of the site.

Sediment surveys in flowing water areas, such as a river, would require a grid system adapted to the longitudinal dimensions of the river.

- where the investigation centres around a point source input of contaminants, a deterministic method is often used, with stations located at intervals downstream of the source. Station location would be based on suitable sediment types, with preference given to areas of similar substrate type in order to reduce variability and enhance comparability of results.
- stations can be points in a river, or preferably, located on transects which span the river from bank to bank. If transects are used, a minimum of three points along the transect should be sampled (one sample in midstream, and one each on either side, the location of which would depend on the channel configuration at base-flow conditions).

b) Station locations should be considered in relation to other potential impacts.

The location of sampling stations in the study area should also take into account the location of existing water intakes and outfalls, the heterogeneity of the bottom materials, and water movement (wave/current action).

- the distance between stations in a deterministic method, or the size of the sampling grid in a random or grid-based method depends on a number of factors, such as heterogeneity of the bottom sediments, the source(s) under investigation and the available funds for the sampling program.
  - in areas of heterogeneous sediment, the number of sampling locations/stations should be larger than in areas of more homogeneous sediment in order to adequately define the sediment and the contaminant distribution.
- c) Spacing of the stations or grids should be based upon the size of the study area.
- where the area studied is large, the grid areas (usually squares or triangles) will also be large. The result is that the area of

sediment each sample represents also becomes proportionally larger. This presents difficulties, since the larger the area each sample has to represent, the less representative of that area each sample becomes. The resolving power of studies based on large grids is usually low, and these studies are most effective when performed in an area of generally more or less uniform bottom characteristics. They would, for example, be suitable for the study of the profundal areas of large lakes where the physical characteristics of the area would not be expected to change over large areas.

d) The physical characteristics of the sediment can influence the number of stations and their locations since sediment type has been shown to significantly affect the distribution of contaminants. For assessment of chemical contaminants, sampling should concentrate in areas of fine-sediment accumulation. The use of a grid in sampling areas that are similar in physical characteristics (i.e., depth, sediment type) can result in the collection of a large number of samples where a few would suffice (Baudo 1990).

- sampling programs designed to assess the nature and extent of contaminants in sediments (from either point or non-point sources) should be directed towards sampling areas of fine sediment accumulation. Fine sediments often accumulate higher levels of contaminants than coarse sediments, since fine organic matter will preferentially bind many persistent organic compounds. Metals are also affected by organic matter through the formation of metal-organic matter complexes.
- in flowing water, preference should be given to areas of fine sediment accumulation, such as natural depressions in the bottom, pools, quiescent areas or artificial depositional areas such as occur behind dams.
- in a random sampling design, stratification based on particle size would be the most

useful. Since stratification depends on preliminary information or existing studies, this technique can only be used where such information exists.

- stratification on the basis of particle size may not be advantageous where the aim is to assess the sediment characteristics of a section of a waterbody or watercourse such as in sediment mapping studies.
- in areas of heterogeneous sediment, the number of stations required to adequately characterize the substrate will be higher than in areas of homogeneous sediment distribution.

For basin-wide or sediment mapping studies, where general assessment of sediment conditions within an area is the primary aim of the study, sampling is usually based on a pre-determined pattern and will often include areas of varying grain sizes.

- in these types of studies it is necessary to sample the existing bottom sediments throughout the area in order to properly characterize the sediments, usually in terms of the existing sediment types and their respective contaminant concentrations.
- the potential biological availability of contaminants from sediments of coarser size fractions (i.e. sands) low in organic matter is generally higher than from fine sediments and their assessment may be important in terms of determining potential remobilization of the contaminant. However, in most cases this sampling should be considered as additional sampling, and not done at the expense of sampling the fine-sediment.
- in flowing water situations, the stations could be arranged on a more regular grid pattern and sampling locations selected either through random selection or through the deterministic method. In such cases, preference should not be given to sampling any one sediment type, since the distribution of sediment types and their contaminant concentrations within the river

is one of the aims of the study. However, such studies would usually require a larger number of samples in order to adequately characterize an area, since both coarse and fine grained sediments need to be characterized. In such cases, prior knowledge of the sediment physical characteristics would be necessary.

- where sediment conditions would be expected to be relatively uniform over large areas, such as in large lake basin-wide studies, the number of sampling locations in the deep profundal areas could be reduced, with relatively large distances between sampling points.
  - where more heterogeneous conditions exist, such as in nearshore areas or in harbours and river mouths, the number of sampling points should be greater and the sampling points closer together.
  - prior knowledge of the sampling area can be a significant asset and can ultimately determine the success of the program.
- e) In areas where the bottom characteristics are variable, the area may be subdivided into smaller study units.
- Sampling in these smaller areas would be based on grids as well, but the grids should be smaller, such that each grid represents a small area of bottom. This will ensure a higher density of sampling within these areas. In nearshore areas where bottom characteristics can be highly changeable, some variant of stratified random sampling can be used.
- f) A suitable control or controls must be located upstream of the study area or outside of the zone of impact. Stations do not have to be placed an equal distance apart and could, for example, be spaced further apart with increasing distance from the source. This would permit higher sampling density in those areas closer to the source, where the greatest impact would be expected. Sampling should be

extended far enough from the source(s) that the final sample lies outside of the zone of impact.

g) The sampling design should consider data requirements for statistical analysis. Where such requirements exist, the design should be modified such that adequate information will be available to carry out the analysis.

- Combining different types of sampling grids usually limits the use of the data for routine statistical tests. However, techniques such as "kriging" are available for analysing such information and are recommended where there is a mix of sampling or grid density. The larger grids can be subdivided into smaller grids, thereby increasing the sampling frequency within an area. Thus, where a harbour or river mouth is located within the larger study area, this area can be sampled at a density greater than the open (profundal) lake areas. Generally, a grid would be sampled in the centre of the grid, though any part of the area can be used as long as this is consistently followed throughout the study. For example, the intersections of the lines could form the station locations.

Many of the methods for determining sampling locations that have been described have depended on existing historical or preliminary data. Often, however, such data are not available and planning must proceed without the benefit of prior knowledge of the site. Under these circumstances the sampling design is usually based on a regularly spaced grid with station locations determined randomly or deterministically. Efforts should be made to sample as many stations as possible, since in most cases the survey will form the baseline study in that area. A large number of sampling points is also essential for any study where statistical analysis forms a part of the data analysis (e.g., trend analysis, GIS, etc).

h) Waterbody Dynamics

The density or spacing of the stations will also depend on the flow dynamics of the receiving water in relation to the discharge. A high volume dis-

charge into an area with pronounced wave or current action, or to a large river with strong flow would carry a larger contaminant load for a greater distance from the source, resulting in a greater area to be sampled. In some cases, sampling may have to be carried out to the mouth of the river, since this is the area where most of the fine sediment load (and associated contaminants) will be deposited.

- The importance of preliminary or historical information in the success of a study cannot be overstated.

i) Contaminant Characteristics

In planning the station locations, consideration should also be given to the type of contaminant(s) involved and the suspended and bed load of the river. Contaminants that sorb rapidly to suspended matter will be carried with this material, while contaminants that remain in solution for extended periods may only be of concern in the lake or other body of water into which the river drains. In standing water, such contaminants may be broadly dispersed throughout the waterbody. In either case, availability of contaminants to biota may be considerably enhanced.

j) Subsurface Sediment

One additional consideration, that will not apply to all types of studies, is determination of the depth to which samples should be taken. The accumulation of sediment over time can result in variations in contaminant concentrations within the subsurface sediment layers. In most sediment assessment studies, only surficial sediment characterization is of concern. However, where a historical record is required, especially where remediation is a concern, or where dredging is proposed, sampling may have to be undertaken to considerable depths.

- depth of sampling is determined by the specific aims of the study and often these are related to assessment of the effects of historical sources of contaminants.
- for dredging projects, where it is often necessary to characterize material to depth,

sediment core samples should be taken to characterize the full depth of the material. Similarly, for lakefilling activities, where there is a possibility of changes in wave and current patterns that may heighten erosion, or where existing depositional areas may become erosional, sediment cores should be taken to characterize the full depth of the erodible material.

- the depth of sampling should be based on an estimate of the yearly sedimentation within the area (harbours will naturally have a higher sedimentation rate than deep basins of large lakes in well forested watersheds) and the historical data available for the operation of any contaminant sources.

Sampling of surface and sub-surface sediments requires different sampling devices as well as different approaches to sampling design. A discussion of sampling devices for sediment studies is provided in Section IV of this report and also in Appendix 2.

#### k) Number of Stations:

The number of stations necessary to adequately characterize sediments within a study area will vary according to the type of study and the aims of the study.

The number of samples required to obtain a statistically significant result has always been a difficult issue to address, since the distribution of contaminants in sediment is essentially non-random. For statistical purposes, characterization of sediment quality at the  $P > 0.05$  level can range into hundreds of samples, depending on the level of certainty desired. Dividing the study area into a number of sampling locations, and collecting replicate samples from each of these has been devised as a practical alternative to collecting a large number of samples at a single station (Baudo 1990).

Baudo (1990) discusses methods to determine the statistically acceptable minimum number of stations for any sediment survey where data are available from previous studies. The procedures can be used to determine the number of stations necessary to derive an average value for an area, with a given statistical uncertainty.

The density of sampling will reflect both the needs of the project and the availability of resources. Thus the amount of detail needed will determine the number of stations.

In most sediment studies, the final aim is to compare sediment contaminant concentrations with the available guidelines.

- where sediment contaminant assessment is the specific aim of the study, the number of replicate sediment samples should be set at a minimum of three. The mean of the replicates is compared to the guidelines values.
- between three and five replicate samples from each station are recommended in order to provide an estimate of the mean and standard deviation around the mean.

Cuff and Coleman (1979) noted that between 2-5 samples per station provided reasonably precise estimates of the mean for benthic samples, based on a stratified random sampling design. Since the distribution of both benthic organisms and contaminants is non-random (i.e., both are significantly influenced by the accumulation of organic matter), this could be applied to sediment samples as well. However, it is recognized that this type of sampling can add significantly to the cost of a study, or alternatively, may lead to a reduction in the number of stations/locations sampled. Therefore, many studies rely on a single sediment sample per station, usually collected as a composite of a larger number of samples.

- composite samples are obtained by collecting a number of replicates (usually 5) which are then combined and homogenized. A sample of the homogenate is collected for analysis.
- for most purposes this will be acceptable, though the sample will give only an average/mean value over that area and, while costs are minimized, the method does entail a loss of information such as the range of contaminant concentrations encountered.

- in cases where remedial action may be considered, or where severe contamination is expected, composite samples are not recommended. In such cases, a larger number of replicates should be considered in order to more clearly and accurately define the nature of the area.

## SECTION II: DEVELOPMENT OF SEDIMENT ASSESSMENT STUDIES FOR SPECIFIC TYPES OF PROJECTS

### 2.1 Lakefilling

#### 2.1.1. Design Considerations

Lakefilling projects involve the placement of fill material in water, generally adjacent to land. Lakefilling activities can result in the suspension of sediment from disposed material and in the dispersal of suspended sediment and associated contaminants.

A sediment survey associated with a lakefilling project is normally carried out by the proponent in reference to one or all of the following objectives:

1. To determine local ambient conditions in order to set an upper limit for fill quality for material suitable for open-water placement (determined according to the procedure described in the *Fill Quality Guidelines for Lakefilling in Ontario*).
2. Set baseline for construction and post-construction comparisons.
3. Determine baseline biological conditions. Such information is used to determine the impacts of lakefills on habitat and estimate the significance of such changes on ecosystem integrity.
4. Determine whether an area is depositional or erosional based on grain size. This information is often essential for predicting changes that could result from a lakefill structure.

In order to achieve these objectives, the survey design should provide adequate coverage of the area.

#### 2.1.2 Survey Design

Surveys conducted in support of lakefilling activities are undertaken to determine existing sediment conditions at the site. This is necessary since the Fill Quality Guidelines require that fill material not be of poorer quality than existing sediments in the receiving area. Details on the assessment of fill quality are presented in the Materials Management Policy and the *Fill Quality Guidelines for Lakefilling in Ontario*.

The survey design would commonly involve the following:

1. Review of existing data with particular emphasis on gaps in the data. The existing bottom characteristics (sediment particle size and contaminant concentrations) should be plotted on a bathymetric chart. This information should be combined with a review of existing biological and water/sediment quality information to determine present conditions in the area. Consideration must also be given to other activities within the watershed area, such as industrial/municipal discharges and urban drainage that could influence changes in the study area.
2. Design of a sediment and biota sampling program, taking into account existing information and in particular directed at filling any gaps in the database. Locations of sampling stations should be plotted on the bathymetric chart. In most cases, only surficial sediment samples will be necessary. However, where it is predicted that changes in wave and current patterns will heighten erosion, or where existing depositional areas may become erosional, sediment cores should be taken to characterize the full depth of the erodible material.
3. Modelling of water movement, particularly as it affects erosion and deposition of sediment materials, should be undertaken to predict effects of altered shorelines on movement of contaminated sediments and identify possible areas of accumulation. Studies should also address the movement of water to ensure that the shoreline alteration will not create

areas of poor water circulation that could lead to degradation of water quality and accumulation of contaminated sediments within these areas.

4. A description of existing conditions at the proposed lakefill site and prediction of potential effects are obtained by combining information from the above three phases. The resulting information should provide an indication of sediment movement and bottom sediment redistribution. Particular attention should be paid to identifying the potential for redistribution of contaminated material and the resultant effects on biota as well as effects on existing and future water uses that may result from the proposed lakefill structure.

In the absence of previous data, the sediment samples should be analyzed for the parameters listed in Tables 1 and 2a of the Provincial Sediment Quality Guidelines. Additional parameters may be required by MOEE on a case-by-case basis. Where additional contaminant concerns have been identified in previous studies, these parameters should also be included in the analysis. The protocols for laboratory analysis for these parameters are available in the MOEE Handbook of Analytical Methods for Environmental Samples (1983).

The suitability of materials for lakefilling depends in part on the background and/or ambient sediment levels in the lakefilling area. Therefore, in any sediment study associated with lakefilling it is necessary to characterize the receiving area sediments.

- To determine background concentrations, a minimum of 5 replicate samples are required from a physically similar area that is removed from all discharges.
- To determine ambient concentrations, a minimum of 5 replicate samples are required from the closest depositional area offshore of the lakefill.

## **2.2 Dredge Disposal**

Dredged-material disposal operations generally fall into one of three main groups; material from maintenance dredging, usually performed in order

to maintain navigational depth in shipping channels and harbours; material from capital dredging for construction of docks, boat slips, turning basins, etc; material from the removal of contaminated materials resulting from in-place pollutants or spills. Since dredging removes material to a certain predefined depth, the aim of the study is to define, both areally and vertically, the chemical characteristics of the material to be removed.

A detailed protocol for dredging proponents is provided in the Ministry of Environment documents "Evaluation of Construction Activities Impacting on Water Resources Part III: Handbook for Dredging and Dredged Material in Ontario".

Under the PSQGs, sediment surveys for dredging projects are carried out for two main purposes: 1) To determine the suitability of the material for various disposal options and; 2) to determine the type and quality of the material at the disposal site. For most dredging programs, sampling points within the area to be dredged can be located using a regular grid pattern, with sampling points either at the intersection of grid lines or in the centres of the squares defined by the grid lines. Sampling should be carried out to the full depth of the material to be removed which will often require depth profiling using core samples.

In areas where sediment contamination may be expected there is an advantage to the proponent to increase the number of sampling sites in order to define as closely as possible the border between material that can be disposed of in open water and material that cannot be placed in open water (see Persaud *et al.* 1992 for detailed protocol for assessing suitability for open water disposal). Where the boundary may lie between two sampling points, all material up to the station where levels are below the L.E.L. would be considered as not suitable for open water disposal. Increasing the density of sampling would serve to reduce the size of the unknown area, with attendant potential cost savings.

### **2.2.1 Design Considerations**

The proponent is required to contact the MOEE Regional office prior to commencing any

work. In all cases design of the sampling program should be undertaken in consultation with Regional staff.

Unless otherwise directed by MOEE, chemical analysis for most dredging projects would entail analysis for the compounds identified in the Provincial Sediment Quality Guidelines.

A careful review of historical data should be undertaken before a sampling program is designed. The data review should consider the following:

1. Does the information meet regulatory requirements?

- Are there results for all parameters of concern for that specific area?
- Are chemical analysis results recent (less than 2 years old)?
- Are analytical methods and detection limits appropriate and adequate?
- Have the data been generated with adequate quality assurance and quality control practices in place?

2. Does the information adequately define the nature of the material to be dredged and disposed of?

- Were an adequate number of samples taken?
- Do the samples represent surficial sediment or provide a complete depth profile of the material to be dredged?
- Were the samples collected and handled appropriately?

3. Are there any long-term temporal trends in the data which indicate a change in the degree of contamination in the project area?

Proponents should be aware that rarely, if ever, can a dredging project proceed solely on the basis of historical data. In almost all cases, current sediment quality results will be required.

### **2.2.2 Summary of Sampling Program Design for Dredging Projects**

Prior to planning, the proponent should always contact the local Ministry office. Project design should only proceed in consultation with Ministry

Regional staff.

**Aim of the Study:** The basic aim is common to all dredging projects - to characterize the sediments in the area to be dredged in order to determine environmentally safe disposal options. The study should always be designed to achieve this aim.

### **Sampling Program**

1. Define area and volume of material to be dredged.

2. Assemble all existing information on sediment quality in dredging area.

- historical data on sediment type (grain size, TOC)
- historical data on sediment concentrations and distribution
- does sampling data extend to full depth of sediment to be removed?
- is data sufficiently current to be of use?
- is data available for all the necessary parameters?

3. If historical data are not available, the sampling program design should include a preliminary survey.

4. Collate existing data on a bathymetric map of the area, identifying gaps in the data.

5. Use all of the above information to determine:

- number of samples required to define sediment quality within the statistical level expected.
- depth of sampling and hence, the type of sampler.
- sampling locations
- parameters (basic PSQG list plus additional compounds based on preliminary information)

6. Collect samples and preserve according to laboratory protocol.

7. Compare results of sampling program to PSQGs and, where high levels have been determined, to Regulation 347 (previously Reg. 309) requirements.

8. Determine disposal options.

sampling program and how the component supports the overall aims of the study.

## **2.3 Sediment Assessment Studies/Surveillance and Monitoring**

### **2.3.1 Design Considerations**

Sediment assessment studies are often included as part of routine surveillance and monitoring programs, usually in relation to a site with known contaminant concerns. Sediment studies are also necessary where preliminary studies have indicated there is an exceedance of the Severe Effect Level of the PSQGs, or where contaminant concentrations exceed a combination of the Lowest Effect Level and the local background. In addition, sediment assessment studies may be performed as part of a spills assessment, a RAP investigation or an Environmental Assessment (EA) or Class EA. Sediment assessment surveys can include any of a number of components such as:

- sediment sampling to determine the extent and severity of sediment contamination.
- benthic sampling to determine whether effects of contaminants are apparent on the sediment-dwelling invertebrate community.
- tissue analysis of resident biota as an indication of contaminant availability and uptake.
- pore-water sampling or geochemical fractionation to determine the potentially available contaminants (to biota and the water column).
- laboratory bioassays to determine any long-term chronic effects of sediment contaminants on aquatic organisms.

The choice of components will depend on the characteristics of each site.

### **2.4.2 Survey Design**

Since sediment assessment involves a number of components, the project should be divided into different phases. For each phase the proponent needs to develop:

1. A clear statement of the objectives of the

2. A definition of the study area. The proponent should assemble all available information on the study area prior to developing the study design.

The data review should draw together all the information available, including:

- known or suspected sources of contaminants. This requires knowledge of inputs to the area such as industrial/municipal outfalls, urban runoff and land use.
- previously available information (i.e., previous studies) on sediment contaminant concentrations and associated biological effects (e.g., tissue residues, community-based effects).
- size of the area/waterbody affected.
- knowledge of existing physical conditions e.g., wave/current patterns, sediment type (depositional/erosional).

Potential sources of information include:

- plant operating records
- government agencies (MOEE, Environment Canada, DFO, etc)
- universities
- municipalities/Regional governments
- consultants

3. Design the survey to address all of the pertinent questions/concerns raised. These would include:

- the areal extent of sediment contamination,
- depth of the contaminated material
- the effect of the contaminants on aquatic biota.

4. Determine which chemical parameters to include. This will depend on the available information for the site.

- the PSQG list serves as a basic starting point. If there are known or suspected sources of contaminants to the area, or multiple sources, then the sampling program can be expanded to include these contaminants. If preliminary work shows that only some compounds are of concern then the study can concentrate on these, provided that the full set of PSQG compounds has been analyzed for during the preliminary work.
  - where contaminants suspected of having adverse effects on biological components both chemical and biological analyses may be a concern (tissue analysis of in-situ biota, community analysis, laboratory bioassays) should be included, with sampling undertaken concurrently.
  - where chemical concentrations are below the Lowest Effect Level of the Provincial Sediment Quality Guidelines, additional assessment of biological effects is at the discretion of the investigator.
  - where concentrations exceed the Severe Effect Level or are close to these levels (i.e., above a combination of Lowest Effect Level and background) the Provincial Sediment Quality Guidelines specify that additional biological sampling would have to be undertaken.
  - for metals, the geochemical distribution between the various sediment fractions may be of assistance in determining the biological availability of metals and may also provide a useful indication as to whether the contaminants are of natural or anthropogenic origins and the length of time they have been in the sediments.
5. Determine the location of sampling stations, taking into account the source(s) of the contaminants and the physical characteristics of the area (i.e. bottom configuration, wave and wind patterns).
- Station location can be based on either a deterministic method or random sampling depending on the aims of the study. Location of stations in a whole lake study, for example,

may be more effectively done on the basis of random sampling especially where statistical analysis is to be used, whereas the assessment of a specific area may be more suitable for a deterministic method.

Note: Where dredging is planned as part of the remediation of a contaminated site, the sampling program should include a sufficient number of stations such that the boundaries of the contaminated area can be defined with some precision. This is to the advantage of the agency/proponent undertaking the dredging. In sediment clean-up operations where the boundary falls between two sampling points, the dredging would have to be to the lowest point. If these are separated by a relatively small distance, the result can be a considerable reduction in the material that has to be removed. Thus, the more precisely the proponent can define the area to be removed, the less extraneous sediment has to be dredged.

### 2.3.3 Summary of Sampling Program Design for Surveillance and Monitoring Studies

1. Clearly define the purpose or aim of the study, listing all sub-aims.
2. Assemble all historical information on the sub-aims.
3. Determine active and historical sources of contaminants.
4. Identify contaminants of concern.
5. Define extent of study area (to include areas outside the zone of impact), incorporating the necessary controls.
6. Define sampling strategy based on:
  - historical data
  - contaminants of concern
  - sediment type
  - specific aims such as depth profile, etc.
7. Determine sampling pattern.
  - random
  - grid
  - deterministic

- stratifying variables
  - number and location of stations
  - number of replicate samples
8. Determine analytical parameters (PSQGs and others, biological parameters).
  9. Choose appropriate sampling device.
  10. Determine sampling schedule (e.g., for biological sampling component).
  11. Collect samples.
  12. Data analysis.

#### **2.4 Spills Assessment and Clean-up**

The design of a sediment sampling program for the assessment of a spill requires some special considerations. Without exception, the aims of such studies are to assess the extent of the contamination and determine the immediate need for remediation. In most cases, clean-up will consist of some type of dredging.

The principal aim is to rapidly assess the extent of the spill. This can be done visually by divers, where it is safe to do so. It is also necessary to assess the depth of the contaminated material, which will require sampling and chemical analysis. Since visual inspection may not necessarily define the extent of the spill, the final sampling area should be at least one third larger than the estimated area of the spill. In any event, it will be necessary to determine ambient concentrations of contaminants outside the spill zone, since these will determine the clean-up criteria.

1. Establish boundary of spill by initial visual survey, if safe to do so. Otherwise, conduct visual survey from boat or sampling platform, visually inspecting sediments, until an approximate boundary can be established.
2. Assess existing physical factors such as current, slope, wave action, that may influence movement of spilled material.
3. Assess the nature of the material spilled (physical and chemical properties)

4. On the basis of 1. to 3. above, determine the extent of the sampling area. The final sampling area should be 1/3rd larger than this area since in all clean-up operations both the extent of the spill and local ambient levels need to be defined.
5. Select most appropriate sampling device. In nearly all cases some cores will always have to be taken since depth characterization will be required to determine proper clean-up depth.
6. Define parameters for analysis. This will be determined on the basis of the material spilled and not the PSQGs.

### **SECTION III: BIOLOGICAL ASSESSMENT OF SEDIMENT CONTAMINATION**

Biological assessment programs are often necessary in order to determine in more detail the effects of contaminants in sediments on aquatic organisms. Contaminant effects can be manifest at both the population level and at the individual organism level. Assessment of the effects at the population level are made through the analysis of community structure, while the assessment of effects at the individual level is most often through analysis of contaminant uptake and toxicity. Benthic communities lend themselves most readily to these types of assessments and are the ones commonly used, though the fish community can and often is used as an assessment tool. The close contact of benthic organisms with sediment, and their relatively sedentary life history, render them particularly well suited to the assessment of sediment conditions.

Biological assessment is generally a major component of any assessment work where sediment surveys have shown that contaminant levels exceed the Severe Effect Level or a combination of the Lowest Effect Level and the local background.

#### **3.1 Benthic Surveys**

The analysis of benthic communities has been used for many decades as a means of assessing the health of a waterbody. While originally applied to

the assessment of the effects of organic matter and the attendant physical effects such as de-oxygenation of the bottom water, it has been shown that chemical contaminants in the sediment and water column can affect the composition and structure of benthic communities.

Analysis of the species composition of benthic communities at its simplest level is based upon the presence of certain species and the characteristic absence of others. The method is founded on the classical ecological definition of a natural, undisturbed community as one that consists of a few species which are commonly distributed and present in greater densities, and a larger number of species that are sparsely distributed and relatively rare (Odum 1966). In a natural state, such a community exists as a balance between these two groups. Under environmental stresses the natural balancing mechanisms are disrupted and an unnatural, stress-defined community or assemblage of species is favoured (Hynes 1960, 1970).

### 3. 1. 1 Community Structure/Functional Analysis

The design of sediment biological sampling programs is the same regardless of the type of project (i.e., lakefilling, dredging, or sediment assessment). While a biological component is usually not required as part of a dredging proposal, biological assessment is often necessary in lakefilling as part of the EA process. Biological assessments are typical components of sediment assessment studies.

Benthic surveys are commonly used as one measure to determine the effects of contaminants on the biological community. The choice of benthic organisms for such studies is based on a number of considerations:

- the organisms are relatively sessile
- a short life history, which makes measurement of effects through many generations relatively easy and apparent
- a wide range of environmental tolerances
- ease of collection

Benthic organisms, even though predominantly sediment dwelling, are exposed to both the water column and the sediments. Thus, they will react to

adverse conditions in both. While they are suitable as indicators of the general health of a system, they usually cannot be used to determine the specific causes, particularly where these may be varied or complex.

### Study Aim

The aim of most benthic studies carried out in relation to sediment studies is to assess the biological integrity of an area in order to determine whether any adverse effects are present due to contaminants in the water column or sediments.

### Study Design

Since sediment type is one of the main factors affecting invertebrate distributions, it is important to attempt to sample areas of similar sediment type in order to ensure comparability among stations and make the results more meaningful. The sampling design methods for benthic sampling are similar to those for sediment sampling.

Benthic sampling has often been undertaken as a stand-alone study (to limited effect), and in these cases stratified random sampling, with sediment type as the stratifying criteria, has been the most commonly used sampling design. This is the most useful method when a variety of substrate types exist within the study area. However, in most cases benthic sampling is carried out in conjunction with sediment assessment, and the design established for the sediment program would also be used for the benthic program.

Assessment of community structure is often used as a means of assessing biological health of the sediment dwelling community, and by inference, of the sediments as well. Assessment can also use analysis of functional feeding groups or other functional indicators.

The majority of insects are seasonal in their distribution in the sediments. Within a species, most individuals will develop at similar rates, and progression through the various life history stages is closely coordinated among the individuals. Thus, during emergence periods, virtually all individuals of a species may be absent from a given area and design of a sampling program should consider such

seasonal factors. Many insects have a summer emergence period, during which time populations in sediment may be reduced or absent. For most assessment programs, the choice of spring sampling, before peak emergence would be best.

### Station Location

Differences in sediment type, water depth/temperature and flow, all affect the composition of benthic communities in major ways. In designing a benthic survey it is preferable to reduce the variability among habitats as much as possible in order to facilitate detection of effects due to the source under investigation. To the extent possible, stations should be located in areas of similar depth, flow velocity and substrate type (grain size, organic content) in order to make inter-station comparisons possible and the analysis meaningful.

In designing a study, a suitable control (i.e. a station in an unaffected area that is the same substrate, depth, etc) to which other stations can be compared is essential.

Equally important in planning a survey is to ensure that all available information is researched. Prior knowledge regarding the distribution of sediment types and any previous benthic surveys would be the most critical pieces of information in addition to known sources of contaminants and loadings.

The number of sampling locations will be determined by the size of the area, the type of effect being investigated and the financial resources available. Since benthic invertebrate distributions are non-random, this will usually require a larger number of samples in order to obtain an estimate of population size (Elliott 1977).

Where contaminant effects are anticipated to be far-ranging within the area, a larger sampling area should be chosen. As is the case with sediment sampling, the larger the area each sample has to represent, the less representative of that area the sample becomes. Similarly, where effects boundaries are important, a larger number of stations should be chosen in order to adequately characterize the boundary area. In many cases, samples can be collected in anticipation of future need and can be stored for later analysis if the need arises.

Since benthic invertebrate distributions are generally contagious, the optimum sampling strategy would be to collect a number of replicates at each location.

### Number of Samples

The number of samples needed to estimate the benthic community to within a 95% confidence interval has been variously estimated as anywhere from 24 to 379 (Resh 1979). As noted earlier, the solution to this problem has been to divide the area into a number of sampling stations. At each location, the number of replicates is commonly set at three to five. Where rigorous statistical analysis is required, Elliott (1971) presents methods to determine the number of stations required to achieve a predetermined level of statistical certainty.

The type of sampler can also affect the result. A large number of smaller replicates can often give a better estimate of the community than fewer larger samples. Studies such as those by Cummins (1975) and Elliott (1971) have found that small quadrat sizes are generally more efficient for sampling invertebrates due to their contagious (i.e., clumped) distribution. Thus, a larger number of core samples is generally more representative than a smaller number of grab samples. However, a large number of core samples is usually more expensive to process than a smaller number of grab samples.

### Sample Collection

The devices used for sediment sampling are also those used in the collection of samples of benthic invertebrates. Grab samples are the most commonly used, though cores may be more suited for certain types of studies (e.g., historical studies of population changes, such as those based on chironomid head capsules).

Due to design characteristics, coring devices generally provide the most representative samples with the least bias, at least in soft substrates. Grab samplers are affected by sediment texture and the size of the sample will depend on how deep the sampler sinks into the sediments and on whether the sampler will rise upon closure of the jaws. Thus, while the surface area to be sampled will be the same, the samplers do not always sample the

same volume of sediment. With gravity coring devices a similar problem can be encountered where the sampler may sink down into the sediment. Core samples are directly comparable only if the volume of the sample can be closely controlled to ensure a similar depth is collected each time. However, grab samples are more cost effective since fewer samples are required to sample the same area of sediment. Benthic samples should not be composited, but rather individual replicates should be collected whenever possible.

In soft sediments, sampling with either corers or grabs requires that the sampler be lowered slowly to minimize the creation of shock waves at the front of the sampler that may disturb and resuspend sediment material and attached organisms, thus biasing the results.

The collection of samples from firm substrates and hard-bottomed areas presents special problems. In deeper waters, firm substrates such as gravel or cobble often require specialized equipment (suction samplers) which generally must be operated by a diver. In shallow waters these can be collected with simple devices such as surber samplers. The mixing of soft and hard substrate areas within a study area can also lead to considerable bias due to the volume of sample collected.

#### Sample Collection Procedure:

- Benthic samples should be washed in the field to remove excess sediment. The sample should be washed carefully to avoid damaging the organisms since loss of taxonomically important structures such as gills can preclude accurate determination and thus can negate the value of the sample.
- Sample residue should be preserved in neutralized (with sodium borate) formalin (37% formaldehyde solution) diluted to a 5-10% solution. The neutralization is necessary to prevent deterioration of mollusc shells by the formalin (most taxonomic keys rely on shell characteristics for species identification).
- For most applications, the mesh size should not be larger than U.S. 30 mesh (595 microns). Where early instars are needed, as in the case of growth or fecundity studies, a

finer mesh size will be necessary for washing the samples in the field.

- Samples should again be washed in the lab to remove excess formalin and sorting should take place under a dissecting microscope. Identification of organisms should be to the lowest practical taxonomic level, which for the major taxa are outlined below:

Insecta- Species or species groups where possible. Minimum level is genus (including Chironomidae).

Crustacea- Species

Mollusca- Species whenever possible

Annelida- Species

- Subsampling is not recommended as a general rule, but where sample size is extremely large subsampling may be necessary. The most common procedure involves spreading the sample evenly over an area, such as the bottom of a tray, and dividing it into a number of evenly sized grids. A number of grid squares (at least 25% of the entire volume) are chosen. In some cases, for example where statistical analysis is planned, a minimum number of individuals may be required, and these considerations should be incorporated into the design of the subsampling strategy.

#### Data Analysis

Benthic data can be interpreted in a number of ways. The data are usually presented as lists of species with accompanying density, expressed on an areal basis. Descriptive methods have traditionally been used, based on known habitat preferences and sensitivities of benthic species. This requires considerable in-depth knowledge, though it does permit the identification of more subtle effects. However, the method is subjective and the addition of statistical analysis can reduce the subjectivity. The types of statistical treatments available range from simple tests such as analysis of variance to complex procedures such as multivariate tests. The range of procedures, together with their shortcomings are

discussed in Elliott (1977), Green (1979) and Legendre and Legendre (1983).

In the past, broad use has been made of indices, such as the Shannon-Weiner diversity index, though as Karr (1987) points out, this approach may be conceptually invalid. Most were developed to deal with the effects of organic matter and attendant deoxygenation, and may be unsuitable for the assessment of contaminant effects. The effects of perturbations have often been shown to consist of changes in the species composition of an area, with replacement of the more sensitive species by those more tolerant of the specific conditions. In most cases this is an effect that is not easily separable from those of organic enrichment and deoxygenation.

In general, the use of these indices to evaluate benthic organism distributions and density is not recommended.

At a minimum, the analysis method should be able to distinguish between impacted and unimpacted areas.

### **3.1.2 Biota Tissue Residue Analysis**

The aim of tissue analysis studies is usually to determine existing tissue residue levels of various contaminants and, if possible, to relate these to concentrations in sediments, water, or both. This provides an indication of both the availability of the compound and its biological pathways. Samples for tissue analysis are often collected as part of assessment studies, especially where remediation may be considered. Their usefulness to direct sediment assessment must be evaluated in relation to the existence of active sources to the water column. Where active sources still exist these will affect tissue residues and will make the task of determining the effects of sediment contaminants more difficult.

The sampling program design would be similar to those for sediment or benthic studies. In most cases, tissue analysis would be a component of the broader sediment assessment study and in such cases, sampling locations should coincide with sediment sampling.

Sample collection should provide sufficient biological tissue sample for the chemical analysis procedure to be performed. Therefore, the proponent should check with the laboratory performing the analysis for the required volume/weight of sample before the work is undertaken. The sampling devices and sample collection procedures used for the collection of benthic samples would also be used for the collection of organisms for tissue analysis.

A number of important considerations must be taken into account when designing tissue residue sampling studies. A major concern is to ensure that the feeding habits and habitat of the organisms are appropriate to the compartment being investigated. Epibenthic organisms, for example, would be exposed more to water column effects than would the sediment in-fauna. Organisms that live within the sediment and that ingest sediment, such as oligochaetes, would be the preferred organisms, since these would be most indicative of uptake and availability of contaminants from the sediment.

Sampling should also ensure that the same organisms are collected and that major groups are not mixed. Therefore, samples collected for tissue residue analysis should ensure that if different organisms, and in particular, representatives of different functional groups are required, that each is collected and analyzed separately. Tissue residue analysis should be restricted to one type of organism to overcome difficulties associated with different rates and modes of uptake and depuration among different types of organisms.

The third consideration is that collection should be done from depositional areas. Not only are these the areas where fine sediments tend to accumulate, but the majority of benthic in-faunal species also occur in such habitats.

Unlike benthic samples collected for enumeration/community analysis, samples for tissue analysis need to be sorted from the bottom debris in the field and the live animals preserved as per instructions from the lab. MOEE protocol involves:

- metal analysis - a minimum of 2 gms are sorted live from the debris, wrapped in plastic film and frozen.
- organic analysis - a minimum of 4-5 gms of organ-

isms are sorted live from the debris, wrapped in hexane-rinsed aluminum foil and frozen.

The proponent should always check with the analytical lab beforehand regarding specific requirements.

In all cases, control samples collected from outside of the zone of impact would be necessary.

Tissue residue analysis is time consuming and expensive and the need for such sampling should be carefully determined beforehand based on the aims of the study. In most cases not all the stations at which sediment samples are taken are sampled for tissue residue analysis, especially where a large number of sediment samples are taken. In such cases, sample locations should be chosen carefully, based on existing information and should consider contaminant concentrations, sediment particle size, and availability of appropriate organisms.

## **3.2 Fish**

### **3.2.1 Tissue Residue Analysis**

The aim of tissue residue studies is usually to determine the effects of sediment contamination through the food chain. Thus, bottom-feeding fish are used for this type of analysis. This type of analysis must be undertaken with the realization that fish are more mobile than invertebrates and that contaminant residues in fish can be obtained from over a wider area. Since fish are water column organisms, they can also acquire contaminants from the water column and thus tissue residues may not be directly related to sediment concentrations. Finally, food can be a major contaminant pathway for fish and potentially more significant than either sediments or the water column, especially in the accumulation of persistent organic compounds such as PCBs. As a result, fish are better for assessing the general availability of a contaminant through a number of pathways, rather than associating the contaminant with a particular source, such as water, sediment, or food.

Bottom-dwelling fish should be collected at the same sites as sediment and biological sampling and would normally only be included in comprehensive

surveys of the effects of existing contamination. They could be used in sediment studies most effectively where active sources no longer exist and only the sediment or sediment-dwelling organisms could act as a potential source.

A number of techniques are used in the collection of bottom-dwelling fish for tissue analysis. The choice of collecting method in large measure depends on the species to be collected and their habitat preferences. Divers equipped with hand nets have been found to be the most effective means of collecting sculpins, which generally prefer rock or cobble substrates. Seine nets, trawls, traps and electro-fishing equipment are generally more efficient for collecting other species, especially in areas of softer substrate.

Fish are stored in plastic bags and frozen until ready for analysis. The analytical lab should be contacted for specific requirements prior to collection of samples.

As with benthic organism tissue analysis, the collection of fish as part of a sediment assessment program is very labour intensive. Since the benefits of such a program are not necessarily applicable to all situations this component is not recommended as standard part of a sediment assessment study, though it may be useful in specific situations.

## **3.3 Sediment Bioassays**

The laboratory procedures for conducting sediment bioassays are described in detail in Bedard *et al.* (1992). The purpose of sediment bioassays is to determine the toxicity potential of contaminants in sediments on a selection of benthic organisms and fish. The biological response criteria include lethality, sublethal growth effects and bioavailability of sediment-sorbed contaminants. As such, they are often necessary components of sediment assessment studies that relate to historical or on-going sediment contaminant concerns. Sediment bioassays are particularly useful in those cases where sediment concentrations exceed the Lowest Effect Level of the Provincial Sediment Quality Guidelines and are required where concentrations exceed the Severe Effect Level. In these cases it is necessary to determine the severity of the biological effects resulting

from these contaminants.

The bioassays use field-collected, whole sediment which is exposed under static conditions in the laboratory using a number of test organisms. Typically, the surficial layer (top 2-5 cm) of bottom sediment is collected, using a suitable grab sampling device. The surficial layer is the most biologically active zone and is the habitat for the majority of benthic organisms. This layer would be representative of those contaminants that have been recently deposited and thus, most readily available.

Field collection of sediment for bioassays requires collection of sufficient sample volume to perform the tests. For the MOEE protocol, 10L of sediment should be collected at each site. In order to obtain such a large volume of sediment, compositing material from several grabs would be necessary. This method does result in a loss of sediment integrity.

Though not all sampling sites from the survey need to be included in this component, those sites included should be selected not only in relation to sources or areas of contamination, but also in relation to a substrate type suitable for the test organisms. Generally a sand-fine sediment mixture (<2 mm diameter particle size) would be suitable for testing using each of the 3 test organisms in the MOEE protocol. Since the field-collected sediment is pressed through a 2 mm sieve prior to testing, those stations where the substrate is comprised mainly of gravel, cobble or coarse sand would be restricted from testing.

Data interpretation involves comparison of the biological responses between the test sediment(s) and control(s). Two types of control sediments are used in sediment bioassays; the negative and the reference control sediment. A negative control sediment is collected from a relatively clean, uncontaminated site which is used in every bioassay, regardless of the study area or the type of contaminants being examined. The negative control sediment is often used as a substrate for culturing purposes. The aim of the negative control sediment is to determine the acceptability of the test, which is based on the average percent mortality of the control animals and must not exceed an approved value, otherwise the test is considered invalid. The health of the test organisms is also assessed using

the negative control sediment.

At each study location it is also necessary to collect a reference control sediment which is collected near or at the study area, but removed from the source of contaminants. When choosing a reference control, due consideration should be given to the suitability of the sediment as habitat for the test organisms. The reference sediment represents background contaminant conditions and is used to measure any biological effects and chemical bioaccumulation that may arise at these ambient concentrations. The reference control sediment should be physically comparable (i.e., grain size, organic content) to the test sediments in order to help discern those biological effects that may be related to physical rather than chemical factors during testing.

Collected sediment samples should be sealed in polyethylene bags with as minimal an air space as possible to reduce oxidation and transported in properly labelled covered plastic buckets at 4°C. The samples are stored at 4°C until ready for use.

The protocol for sediment bioassay testing is described by Bedard *et al.* (1992) and should be referred to for details of the procedure.

## SECTION IV: SEDIMENT SAMPLING FOR CHEMICAL AND BIOLOGICAL ASSESSMENT

### 4.1 Sampling Devices

Over the year a variety of sampling devices have been developed for sampling sediment and sediment-dwelling organisms. These devices fall into two main groups: grab samplers and core samplers.

Grab samplers are jaw-like devices designed to collect surficial sediments by scooping out a defined area of the sediment surface. The depth of collection is limited by the height of the sampler (i.e., the volume) and the nature of the sediment material. Their ability to collect a sample is a function of the degree of penetration (firmness of the sediment relative to the weight of the sampler), angle of

penetration, depth of water, and lateral motion of the boat or sampling platform during collection. Unless sealed at the top, there is also a tendency for "washout" of fine-grained materials during retrieval.

Core samplers are usually tube-shaped devices which can penetrate the sediment by gravity (free-fall), vibration or hydraulic pressure (water or oil). These collect sediment to a much greater depth than grab samplers (depending on the length of the collection tube fitted to the sampler). Appendix 2, taken from Sly (1969), describes the various types of samplers, both corers and grabs, and their advantages and limitations. Figures 1.1 and 1.2 illustrate the various types of grab samplers and core samplers respectively.

The distribution of contaminants in sediments varies both horizontally and vertically. Horizontal variation can be assessed by the collection of samples from selected sites throughout the project area. Typically, the concentration of contaminants also varies with depth in sediments. If information on variability with depth is required, it is recommended that sediment samples be collected using either a coring device from a boat or having a diver collect a core. A grab sampler is recommended if information on surficial sediment only is required.

The typical coring device is a length of pipe with a weighted head of 50 to 200 kg. Inside is the plastic liner (polybutylacrylic plastic is recommended). At one end is a metal core cutter which assists the coring device to penetrate the sediment and a core catcher to retain the sediment in the liner. At the top end is a ball-valve or piston which retains the sediment in the liner when the device is pulled back out of the sediment.

There are three major drawbacks to the gravity core sampler:

1. There is a "shock wave" ahead of the sampler before it penetrates the sediment. This may displace the very unconsolidated top layer of sediment;
2. The gravity action tends to compress the sediment during penetration, thereby compressing the vertical profile of the contaminants (Baxter

*et al.* 1981); and

3. The use of a small sampling boat necessitates the use of a small core sampler with a small head weight. The small barrel diameter of the sampler can cause gross disturbance of the sediment profile during penetration, potentially destroying the vertical profile of the contaminants. The small weight may lead to insufficient penetration.

A diver-collected core is preferred over a core collected by a free-falling coring device. The diver is able to carefully insert the liner in the sediment, minimizing the disturbance of the surficial sediment, virtually eliminating the compression problem and is able to use a relatively wide diameter liner. The use of a diver also permits an observation of the general nature of the bottom and the presence of aquatic biota. A limitation of the diver-collected core is that the retained length is typically less than 1 m.

In very specialized cases, where it is important to preserve the fine structure of the sediments, a large box-corer or hydraulic corer is recommended. The box-corer, because of its size, can collect a sediment sample with the centre undisturbed. A major drawback to the device is that it is large and complex to operate and collects a tubular core similar to the gravity corer. One advantage is the slow penetration which reduces the compression effects noted with the gravity corer.

The core and grab samplers described above are best used in fine sands or muds. Collection of grabs of coarse sand or cobble requires a large and adequately weighted grab (larger than 0.5 sq m capacity). A vibra-corer is required to collect a core in coarse or compacted sand. This device is similar to the gravity corer, but a vibration source is attached which vibrates the barrel down into the sand. Such a corer may require a specialized sampling boat, due to the weight of the equipment and the power requirements.

Final selection of the sampling device will depend on the characteristics of the site and the objectives of the study. Where stratification of the sediment is suspected or is a concern, a coring device would be the preferred choice. Where sediment layers are homogeneous, or the vertical profile of sediment

concentrations is not important to the aims of the study, a grab sampler may be more effective/efficient (in terms of costs).

## 4.2 Field Record Keeping

### 4.2.1 Positioning

Sample site locations should be determined as accurately as possible in the field and precisely located on a map. Positioning is especially important if the sites are to be re-sampled at a later date. Accurate positioning is also important for later analysis of the data using Geographical Information Systems, and where possible should include either geographical coordinates or UTM coordinates.

The sample sites can be determined using landmarks, actual measurements, distance estimator or electronic positioning equipment. When available, electronic positioning equipment (side-scan sonar, Loran-C) provides the most accurate result.

For certain types of studies, such as those for dredging projects, submission of a detailed plan of the project site delineating the site boundaries and the location of the sample sites, is a very important feature of the application for environmental review. A chart scale of 1:500 or 1:1,000 is recommended.

### 4.2.2 Field Notes

The information gathered for sediment evaluation (chemical and biological components) should include field notes covering the following points:

- current speed near bottom
- weather conditions;
- time and date of collection;
- positioning information;
- type of sampler used;
- name of sampling personnel;
- notation of odd or unusual events which occurred during sampling (e.g., "corer returned only a few rocks");
- field description of samples:
  - odour,
  - approximate particle size,
  - colour,
  - presence of non-decomposed organics (e.g., wood fibres),

- presence of oil and grease,
- presence of distinct layering as given by changes in colour or particle size,
- presence and type (to broad groupings) of aquatic biota, and
- length of retained core;
- brief description of handling procedures and types of containers used;
- notation where there was a deviation from standard handling and splitting procedures; and
- laboratory to which samples were delivered and the date of delivery.

If the proponent is routinely having samples collected, a standardized form covering the field information is recommended.

### 4.2.3 Physical Analysis

Before a sample is mixed and split in the field, the odour and colour should be noted and the pH and redox potential measured. Odour can be divided into four categories:

- Odourless
- Chemical
  - chlorine
  - petroleum
  - medicinal - phenol, iodine
  - sulphurous
- Decaying Organic
  - manure
  - sewage
- Natural
  - earthy
  - peat
  - grassy
  - mouldy

Colour can be best determined by comparison of the sediment to the Munson colour code system. If that is not available, each colour zone or depth of core should be described. Colours will range from reddish-brown to jet black.

The pH and redox conditions should be measured with appropriate electrodes which have been properly calibrated. The electrodes should be rinsed with clean water between measurements and

stored in appropriate containers. Accuracy of measurement should be  $\pm 0.1$  pH units;  $\pm 10$  mv for redox potential.

### **4.3 Field Storage and Handling**

#### **4.3.1 Grab Samples**

If redox and pH measurements are required, then the probes should be inserted into the sediments (3-5 cm), as soon as the grab is on-board. If possible, the probes should be inserted and samples removed through top-access doors rather than transferring (and thereby mixing) the sample into a pan. Observations should also be made at this time: presence of oxidized surface layer, colour and smell of underlayer, approximate particle size description and presence of obvious oil or grease or non-decomposed organics (e.g., wood fibres).

The top 3-5 cm of the grab sample should be transferred into a clean pan and thoroughly mixed using a large, clean teflon or ceramic spoon. Subsamples should be handled as follows:

1. For metals/particle size/carbon/ phosphorus/total Kjeldahl nitrogen/loss on ignition, place in clean plastic or glass containers; and
2. For trace organics/oil/grease, place in clean solvent-rinsed glass bottles with clean aluminum foil cover caps. Amber-coloured bottles are preferred.

The amount of sediment required for analyses should be determined in consultation with the analytical laboratory. The samples must be kept at 4°C and out of sunlight. Samples should be shipped to the laboratory as soon as possible after collection. Sample containers should be carefully labelled with indelible ink pens. Labels should contain the following information:

- date and time of collection,
- identification of collector, and
- site identification (including harbour name).

This information should correspond to information recorded in the field notes.

#### **4.3.2 Cores**

With the bottom end of the liner securely capped, the excess water should be carefully decanted or siphoned. The core may need to stand for some time to permit settling out of disturbed material before decanting.

The length of retained material should be measured in centimetres. Excess core liner should be cut off and the top of the liner capped. The core should be retained upright and carefully labelled. It is suggested that the label be placed only on the top end of the liner, to ensure that the core is not inadvertently turned over during transit or storage. The core should be handled in such a way as to prevent "sloshing" of the material.

As with the grab samples, the core samples should be stored at 4°C. As the cores may be long and cumbersome, it may be convenient to split the cores in the field. (This is best done on shore). Before extrusion, the core should be examined and the depths where redox discontinuities occur should be noted. The cores should be extruded from the bottom end (the firmer end). The core sample may be sectioned in one of two ways. The first way is to section the sample according to the different layers if the colours are obvious. Otherwise, samples may be sectioned into top, middle and bottom sections or sectioned at regular intervals (e.g., 5 cm). The actual amount should be determined in consultation with the analytical laboratory. Each section should then be treated as a separate sample and handled as described above. This will include measurements of pH and redox, noting colour, odour, redox discontinuities, approximate particle size and presence of oil or organic matter; non-decomposed organics etc.

#### **4.3.3 Field Storage and Handling where samples will be subject to Partial Geochemical Leaching**

It is critical that the samples not be exposed to air, as geochemical changes may occur. As soon as the grab or core sample is brought on board, the sediment should be placed in a nitrogen-filled bag or glove-box. All sub-samples should be purged with nitrogen and maintained at 4°C. The samples should not be allowed to dry out or be frozen.

#### 4.3.4 Laboratory Handling and Storage

There should be sufficient facilities in the analytical laboratory to store all of the samples at the appropriate storage temperatures. As soon as the samples enter the laboratory, they should be logged into the laboratory sample management system and labelled with the laboratory control number. It is expected that the laboratory undertaking the work will have shown evidence of good laboratory practices (i.e. adequate quality control and quality assurance). Care must be taken to ensure that the samples are not contaminated by other samples in the laboratory.

#### 4.3.5 Archive and Duplicate Samples

Sediments may be heterogenous and therefore must be thoroughly mixed before they are sub-sampled. Each container should be mixed and sub-samples taken for the required analysis. Remaining material should be combined into one container and this preserved frozen as an archive sample. This should be retained for at least one year or until the dredging operation or study is completed.

The purpose of the archive sample is to permit subsequent re-analysis for a particular constituent or external audit analysis. For field and laboratory quality control and quality assurance, the following duplicates must be taken. For example, if 5 samples are to be taken, 1 additional sample is to be taken as a field duplicate. The laboratory views these as 6 unknowns and therefore there would be 6 laboratory samples and 1 laboratory duplicate for a total of 7 samples for analysis.

#### 4.3.6 Sample Splitting Report

To assist the Ontario Ministry of the Environment in their review, the proponent is requested to submit a sample splitting report. This report should contain the following information:

- pH and redox potential measurements (if requested);
- odour;
- visual description of particle size;
- presence of redox discontinuities (e.g., oxidized surface layer), if core, note the depth of discontinuity layers;

- presence of oil and grease;
- presence of non-decomposed organic matter (e.g., wood fibres);
- length of retained core;
- who collected, handled and split the samples; and
- deviations from splitting and handling routine as outlined above.

## SECTION V: SAMPLE ANALYSIS

There are several manuals available which detail methods of analysis for sediments. As with sample collection, analytical methods are common to all sediment related activities and are not discussed under separate headings. The recommended procedures are described in OMOE (1983), *Handbook of Analytical Methods for Environmental Samples* (2 Vol.), Ontario Ministry of the Environment, Toronto. The use of other procedures should be first verified with MOEE labs.

Analyses for chemical determinations (metals, nutrients, organics) should be made using a method that achieves the lowest detection limit.

### 5.1 Particle Size Analysis

The detailed particle size of the sample should be determined for the size range of -4 phi (16mm) (Wentworth Scale) to +9 phi (0.002mm). The determination between -4 and +9 phi may be made using a variety of techniques, these include: sieve, pipette and microtrac (used by MOEE). Percent gravel, sand, silt and clay should be calculated from the detailed particle size analyses.

### 5.2 Trace Inorganic Chemical Analysis

#### 5.2.1 Nutrients

The guidelines require the determination of total phosphorus and total Kjeldahl nitrogen. The methods of analysis are outlined in Plumb (1981) and OMOE (1983).

## 5.2.2 Trace Metals ("Bulk Metal")

The Provincial Sediment Quality Guidelines specifically include the following metals: arsenic, cadmium, chromium, copper, iron, lead, manganese, mercury, nickel and zinc. The metals are analyzed using dissolution in aqua-regia (i.e., hydrochloric : nitric, 3:1 acid digestion). The MOEE guidelines were developed with metal concentrations obtained using the aqua-regia acid digestion. All of these are routinely determined by atomic absorption spectroscopy or plasma emission spectroscopy with due consideration for chemical interferences and detection limit requirements and capabilities.

## 5.2.3 Trace Metals (Partial Leaching)

The use of bulk metal analysis simply characterizes the metal composition of the sample, but does not differentiate the geochemical distribution within the various sediment phases with which the metals are associated. The technique of partial chemical leaching can be used to describe the geochemical phase distribution of the metal(s) and has been used to provide an estimate of the bioavailability of the metal. The techniques developed are strictly method dependent and the results operationally defined. The methods are typically applied to cadmium, copper, lead, zinc, chromium and nickel. The following procedure has been used extensively by MOEE and for comparability it is very important to follow the procedure as outlined and not other sets of sequential leaching procedures. Steps 1-3 must be done in a nitrogen-glove box or bag (i.e., under inert gas):

- Step 1 ● **Interstitial Water** - Centrifuge the wet sediment at 15000 rpm. for 10 min. Decant and analyze supernatant. Sieve the material through a 64 micron sieve (i.e., only the silt-clay fraction).
- Step 2 ● **Cation and Adsorbed** - Shake the wet equivalent of 0.5 g dry weight of sieved material from stage 1 with 1M ammonium acetate solution for 2 hr. Centrifuge at 15000 rpm. for 10 min. Decant and wash sediment with distilled-deionized water; centrifuge and add rinse water to original solution. Acidify to pH 2 and store for subsequent analysis.
- Step 3 ● **Easily Reducible** - the residual sediment from stage 2 is shaken for 2 hr. at room

temperature with 20 ml of 0.1M hydroxylamine hydrochloride at pH 2, centrifuged at 15000 rpm. for 10 min. and decanted. The sediment is rinsed with distilled-deionized water, centrifuged and the rinse added to the original decanted solution. Store for later analysis.

- Step 4 ● **Organic Complexed** - The residue from stage 3 is shaken for 1 hr. at room temperature with 20 ml of hydrogen peroxide acidified to pH 2 with nitric acid, then for 5 hr. at 95°C. Then 10 ml of 1M ammonium acetate (pH 2) is added and the extraction continued for 1 hr. The suspension is centrifuged at 15000 rpm. for 10 min. and decanted. The sediment is rinsed with distilled-deionized water, centrifuged and the rinse water added to the original decanted solution. Acidify the solution to pH 2 and store for subsequent analyses.
- Step 5 ● **Moderately Reducible** - The residue from stage 4 is shaken for 2 hr. at 95°C with 1M hydroxylamine hydrochloride/glacial acetic acid (1:1 volumes) solution. The suspension is centrifuged at 15000 rpm. for 10 min. and decanted. The sediment is rinsed with distilled-deionized water, centrifuged and the rinse water added to the original decanted solution. Store for subsequent analysis.
- Step 6 ● **Residual (Lattice)** - 1.0 g of dried material from the above is dissolved in 3:1 aqua regia following the method for "total" metal.

Calculations should be made of the relative percentage of total metal in each phase.

## 5.2.4 Other Inorganic Compounds

The PSQGs do not specifically refer to cyanide. However, the Open Water Disposal Guidelines did refer to cyanide and in the absence of new guidelines, analysis for these are carried over. The analytical methodology is a water extraction of the sediment, with distillation of the extract, followed by a colorimetric determination using a pyridine-barbituric acid reagent. The method is outlined in OMOE (1983).

Other inorganic compounds (e.g., organo-leads)

may be required to be determined as a result of MOEE Regional Office review. Appropriate methods of determination will be provided by MOEE at that time.

### **5.3 Trace Organic Chemical Analysis**

There is an extremely large number of trace organic compounds that are of environmental concern. Many of these compounds may bioaccumulate and are suspected or known carcinogens or carcinogenic precursors. The Provincial Sediment Quality Guidelines specifically identify PCB with an acceptable limit of 0.07 µg/g in sediment. However, with the increasing evidence that sediments in many areas of the Great Lakes and the Inter-connecting Channels are heavily contaminated with a wide variety of trace organics (e.g., Report of the Niagara River Toxics Committee, October 1984), the numerical guidelines for the evaluation of open water disposal have been expanded to include other substances.

#### **5.3.1 PCB and Organochlorine Pesticides**

The sample is extracted with distilled-in-glass acetone and dichloromethane, concentrated, and the PCB fraction separated from the pesticide fraction by liquid chromatography using for example, a Florisil column. The extracts are reduced in volume, cleaned with solvent-washed mercury to remove sulphur compounds and analyzed using gas chromatography with an electron capture detector. Calibration should be made using standards of the pure compounds. Quality assurance should be monitored by testing the extraction efficiency, the separation of the Florisil and the use of standard reference materials. The method is detailed in OMOE (1983). The procedure can be used to determine: PCB, BHC isomers, Chlordane (both isomers), DDD, DDT, DDE, Aldrin, Dieldrin, Endosulfan (isomers I, II and sulphate), Endrin, HCB, Heptachlor, Heptachlor Epoxide, Lindane, Mirex, and Methoxychlor.

#### **5.3.2 Polyaromatic Hydrocarbons (PAHs)**

This is a particular sub-group of the aromatic fraction of petroleum hydrocarbons. These compounds are produced by the incomplete combustion of coal, coke, petroleum hydrocarbons and various aromatic compounds. Some of these compounds

are considered to be carcinogenic or carcinogenic precursors.

The Provincial Sediment Quality Guidelines do not require analysis for PAHs on a regular basis though guidelines have been developed for most of the compounds listed below. However, the proponent may be requested by MOEE to determine the concentrations of any or all of the following PAHs in sediments where there is cause to suspect PAH contamination:

acenaphthylene  
acenaphthene  
anthracene  
benzo[a]anthracene  
benzo[a]pyrene  
benzo[b]fluorene  
benzo[g,h,i]perylene  
benzo[k]fluoranthene  
chrysene  
dibenz[a,h]anthracene  
fluorene  
fluoranthene  
indeno[1,2,3-cd]pyrene  
naphthalene  
pyrene  
phenanthrene

The sample is solvent extracted in a Soxhlet system, reduced in volume, cleaned up and analyzed by high pressure liquid chromatography with fluorescence and UV-Vis detectors. Calibration is made against appropriate standards of the pure chemicals. The National Research Council of Canada will shortly be offering for sale four marine sediment reference materials, each spiked with selected PAHs which together will provide analysts with a range of concentrations for the 16 PAHs listed above.

#### **5.3.3 Other compounds**

Analysis for additional compounds may be required after MOEE review. The methods of determination will be provided at that time by MOEE.

### **5.4 Miscellaneous Analyses**

#### **5.4.1 Organic/Inorganic Carbon**

This category is often labelled as "loss of weight

on ignition" or total organic matter. There are two principal techniques used: combustion in a muffle furnace at fixed temperatures ("loss of weight on ignition"); and, combustion in a high temperature furnace in a stream of oxygen with determination of the resultant carbon dioxide (furnace method). In both techniques, the dried sample is heated to a specific temperature and the resultant weight or carbon dioxide generation can be quantified to represent total carbon. The other sub-sample is treated with hydrochloric acid to remove the carbonate carbon, and the treated sub-sample analyzed as above with this fraction defined as the organic carbon. The difference is taken as the inorganic carbon fraction. Both procedures are detailed in OMOE (1983).

#### 5.4.2 Solvent Extractables (Oil and Grease)

This is a catch-all category which includes both petroleum hydrocarbons as well as fats, waxes and greases. The wet sample is extracted with dichloromethane and the resultant extract is solvent evaporated and the residue weighed (gravimetric method). A detailed procedure is given in OMOE (1983).

A major drawback is that the method is non-specific. Most non-halogenated hydrocarbons are extracted and the detection method does not offer any means of discriminating between the various hydrocarbon types. The present guidelines specify a limit of 1500  $\mu\text{g/g}$  dry weight. Typically this is taken as total solvent extractable matter. Obviously, significant quantities of non-petroleum hydrocarbons (e.g., plant lipids) will give a "high" value which cannot be directly correlated with lethal or sublethal effects due to petroleum hydrocarbons which might be present.

## SECTION VI: QUALITY ASSURANCE/QUALITY CONTROL CONSIDERATIONS

### 6.1 Introduction

The "quality" of data is a function of the uncertainty of the data compared to its end-use requirements. Thus, data can be acceptable under one

requirement and be unacceptable under another. Quality assurance is the mechanism by which data are produced to meet a defined standard of quality with a stated degree of confidence. Quality assurance quantifies the uncertainty associated with reported data. Quality control is the series of activities used to obtain and maintain that standard.

Quality assurance and quality control (QA/QC) are important elements in all facets of a project. They are mechanisms whereby the proponent can monitor the performance and results of his staff or contractors, and they permit the regulatory agency to determine the quality of data submitted as part of a project review. The complexity of environmental data and the need for comparability has led to requirements for quality assurance and control in the analytical laboratory without necessarily recognizing that quality assurance and control must be applied throughout the program. For example, poor sampling or sample handling practices can obviate the most careful laboratory analyses.

QA/QC should not be considered as just another requirement by the proponent; it should be recognized that it is in the proponent's best interests to provide quality data, since it is that data which will be used to determine the effectiveness of the project.

There are five basic elements in QA/QC:

- completeness of the data set;
- representativeness of the data;
- comparability of data;
- validity of identification; and
- accuracy and reproducibility of quantification.

Completeness of the measurement can be defined as obtaining the amount of data that is necessary to meet the project objectives.

The representativeness of the data is the degree to which data accurately and precisely represent the concentrations of the constituents or the characteristics of the material. For example, a grab sampler samples the top 10 to 15 cm of sediment. If a 1 m dredge cut is to be made, then the sample represents only the top 10 to 15% of the material to be dredged.

The comparability of data is defined as the

degree of confidence with which one data set can be compared to another or to guideline concentrations. This requires that proponents use consistent and documented methods throughout the data collection. Recognizing that sampling and analytical methods are constantly being changed and improved, the Ministry may accept non-standard procedures after consultation with the proponent.

The validity of identification is important for environmental samples where the analyst is asked to determine low concentrations of contaminants in complex matrices. This is particularly true for trace organic compounds (e.g., PCB).

The accuracy and reproducibility of quantification are the elements which most people use to define QA/QC. Quantitative measurements are only estimates with a stated degree of probability of uncertainty. Measurements must be made in a sufficient number and way so as to provide a statistically acceptable definition of the degree of confidence. This will require the analysis of a number of replicate samples by the analytical laboratory and the analysis of standard reference materials. The results of these additional analyses should be included with the application.

## **6.2 Elements of QA/QC Program**

The basic elements of a QA/QC program include:

- technical competence of staff;
- suitability of facilities and equipment;
- good measurement practices;
- standard operating procedures;
- special operating procedures; and
- good documentation.

Good measurement practices can be defined as those procedures which have been tested and refined so that the results are consistently accurate and precise. Such practices can range from ensuring that equipment is routinely maintained and calibrated to procedures for the cleaning of sample containers. Typically such procedures are not documented but are assumed; unfortunately because they are not documented, such practices are variable even within a department.

Standard operating procedures are those procedures which specify how samples are to be col-

lected, handled and analyzed. Such procedures should include the necessary information so that the techniques used can be repeated by another group or laboratory. Similar to the good measurement practices, standard operating procedures are typically not documented except where a laboratory is required to use a method published in a laboratory manual. Such operating procedures must include the procedures necessary to calibrate the techniques or to position the sampling equipment.

Most laboratory procedures are documented and would therefore be classified as standard operating procedures. However, sampling and sample handling procedures are often not documented and therefore special operating procedures should be developed and documented. These procedures should be developed with the assistance of well-qualified staff to ensure that all relevant points are included.

In each stage of the QA/QC, documentation of the procedures and techniques is important. It is in the proponent's best interests to ensure that all procedures are carefully and thoroughly documented, including obtaining relevant documentation from the laboratory. Full documentation will also ensure that the same techniques are used each time samples are collected and analyzed, thus permitting comparison of the data.

## **6.3 Implementation of QA/QC Program**

### **6.3.1 Sampling**

This component is the most difficult to monitor for QA/QC. The proponent must ensure that samples are collected using acceptable equipment and at the specified locations. A check on equipment can be made by the collection of samples from a relatively non-contaminated area. Equipment must also be maintained in good condition and kept clean of obvious contaminants such as rust and oil and grease. Sample site positioning is very important, particularly when sampling at close intervals or if the site is to be re-sampled at a future date. Specialized positioning and survey equipment may be required.

Suitability of facilities and equipment includes the sampling equipment, the vessel, the positioning

equipment, the sample handling equipment and the laboratory facilities. It is in the interests of the proponent to ensure that a grab or core sampler of suitable size and design is used to collect an adequate sample. Due to the weight of such equipment, winches and a boom of suitable capacity will likely be required.

### 6.3.2 Sample Handling and Processing

The proponent must ensure that the samples are transferred from the collection equipment to the laboratory while maintaining the integrity of the sample, limiting contamination and ensuring that there is minimal loss of constituents. It is recommended that samples be transferred as soon as possible from the sampling device to transfer containers. These containers must be clean. Plastic containers should be used for sub-samples intended for trace metal, particle size and miscellaneous components; glass containers should be used for sub-samples intended for trace organic analyses. One container of each type and set should be retained and tested as a sample container blank.

If samples cannot be processed and analysis started within 48 hours of collection, they may be stored at 4°C for longer periods.

### 6.3.3 Laboratory Analysis

In addition to the requirements for suitable facilities and personnel, specific components in laboratory analysis include:

- sample receipt;
- sample processing;
- control charts;
- standard reference materials;
- non-standard reference materials;
- replication; and
- external auditing.

Sample receipt should include maintaining proper documentation, chain- of-custody and ensuring that the specific analytical requirements are defined by the client. A proper logging-in system with some type of laboratory identification is a prerequisite.

Sample processing can introduce errors due to the heterogeneity of sediment. Samples should be

well homogenized before sub-sampling. To assess sample processing quality, split samples should be taken from 10% of the samples and handled as additional unknowns. An archival sub-sample should always be taken and maintained frozen for at least one year. A suitable area should be maintained for sample processing and great care taken to avoid cross-contamination.

Control charts are critical to assessing the performance of the analysis stage. Control charts should be maintained for calibration functions, extraction/digestion efficiencies, reagent blanks and for determining the reproducibility of a measurement process. Control charts can be constructed by the use of repetitive analysis over a period of time of a particular sample or set of samples. In many cases, standard reference materials are used; however, any well-homogenized sediment can be used. While the latter type of materials cannot be used to monitor bias, the initial concern should be to maintain consistency and reproducibility. Each chart should consist of the mean and difference of duplicate determinations with warning and control limits set at two and three standard deviations respectively. The use of control charts should be an ongoing task and should be monitored by the supervisory analyst to ensure that a group of analyses does not go "out of control".

The concurrent analysis of standard reference materials provides both a monitor on bias in the analysis and provides a source of data for the control charts. A variety of sediment reference materials are now available from both the National Research Council of Canada and the U.S. National Bureau of Standards. A limited number of standard reference materials for other matrices (e.g., biota) are also now becoming available. The major drawback is that most of these materials are for trace metals; very few are available for trace organics. It is recommended that two reference materials be concurrently analyzed with each set of twenty samples. Results of analyses should be included with the application.

The proponent may also want to audit the analysis by the use of non-standard reference materials. However, care must be taken to ensure that:

- the materials are similar in matrix and con-

- centration range to the unknowns; and
- the samples are submitted as unknowns to the analyst, so that they are truly "blinds".

There are a number of sources of non-standard reference materials; however, their major drawback is often poor characterization. Replication is an easy method of maintaining control charts and monitoring QA/QC. It is recommended that 10% of the samples be concurrently analyzed in duplicate and that this data be included in the application.

External auditing is also useful as a monitor of QA/QC. This can include participation in inter-laboratory comparisons and participation in contracted analyses of reference materials. Use of outside experts (e.g., university research scientists, National Water Research Institute) to review operating procedures and control charts is also useful.

## SECTION VII. DATA INTERPRETATION

Data interpretation should seek to ensure that the conclusions derived from the results of the study are consistent with, and support, the initial aims. For most sediment studies, the primary goal of data interpretation is to establish the existing sediment conditions with respect to sediment physical parameters, chemical concentrations of contaminants, the composition of the benthic community, and the contaminant burdens of individuals within the benthic and fish community. If concentrations of certain contaminants are found to be anomalously high in relation to adjacent areas, or if biological analysis indicates that there is evidence of environmental stresses, the interpretation should seek to determine the sources, and quantify the biological effects.

Data interpretation should proceed through a number of steps.

1. compare results among stations
2. compare results to established guidelines
3. compare patterns of contamination
4. establish linkages to sources
5. establish linkages between chemical concentrations and biological effects

The results of most data interpretation are not definitive and the conclusions are mainly drawn on the basis of the weight of evidence that supports the conclusions. In many cases a number of supporting tests or analyses may be required and, as such, conclusions should be based on a combination of the above approaches.

1) The first step in data interpretation consists of comparison of results among stations. By comparing results from impacted areas with unimpacted areas, the relative degrees of contamination can be determined. This type of data interpretation is suitable for both chemical and biological components.

Differences or similarities can be evaluated using:

- i descriptive analysis, including graphs, etc.
- ii statistical analyses
  - simple statistics
  - multivariate analyses
- iii indices, usually for benthos
- iv mapping (e.g., GIS) to plot contours, areas of effect, hotspots, etc.

i) Descriptive analysis. Descriptive analysis encompasses all narrative analysis that compares or contrasts the chemical and biological findings among stations. It excludes statistical analysis which is not narrative.

- Narrative analysis of results. For sediment, this generally consists of a description of chemical concentrations in sediment, noting high and low concentrations, their locations within the study area, and their relationships to physical features (e.g., substrate type, depth, temperature). For biological results such as benthic communities, this would involve noting the species present, their ecological role, etc.
- Comparison with other areas/localities. Results are compared with other stations within the sampling area or with stations in other similar localities.
- Graphical analysis (presentation of findings using graphs, etc) noting distribution in the study area in relation to physical characteristics and also their relationships to sources.

ii) Statistical Analysis

A number of statistical techniques are suitable for analysis of chemical, physical and biological information.

- simple statistical tests such as t-test, ANOVA. Stations are compared and, where suitable data is available, differences in means can be tested using these statistical tests. Since in most sediment studies the data distribution is seldom normal, statistical analysis will usually require either non-parametric tests or data transformation.
  - multivariate analyses (e.g., Principal Components, Factor Analysis, etc) can be used to relate variables among a large number of stations. Usually used to group stations that are similar on the basis of the test variables selected. Validity of the analysis depends on the variable selected. Relating chemical parameters to benthic organism distributions is difficult, since a variety of physical factors can influence the density and distribution of benthic organisms. Since the same factors can affect the accumulation of contaminants (i.e., particle size and composition) these can render interpretation difficult and may even result in misleading conclusions.
  - suitable statistical analyses are described in Elliott (1977) and Legendre and Legendre (1983).
- iii) Indices. A variety of numerical indices have been developed to interpret biological information, particularly the results of benthic organism community analyses. These have seen considerable use in the past though they have currently fallen into disfavour. Most are based on an equation calculated on the basis of the numbers of species present and the numbers of individuals of each species. Typically such indices involve the reduction of the data to numerical values and thus entail some loss of information. Most reviews have found that such approaches are of limited value and that often the indices are unable to identify where changes have occurred, particularly where these are not pronounced/gross changes. In addition, there is some question as to the conceptual validity of this approach (Karr, 1987).
- iv) Mapping. One of the greatest aids to interpreting contaminant distributions within an area is through plotting the distribution of sediment contaminants on a map. Typically, though not necessarily, this involves the use of computer systems (e.g. GIS). Most of the programs available (e.g., SPANS, RAISON) will plot data and, by making use of built-in algorithms, will draw contours, etc. This type of analysis will find extensive use in dredging, lakefilling and sediment assessment studies where intensive sampling is involved and areas of sediment to be removed or adversely affected need to be defined.
- 2) Comparison of results to established guidelines. In many cases, studies will be undertaken to evaluate the suitability of sediments for specific uses such as open water disposal of dredged material or for lakefilling. These will require comparison of the results with established guidelines. Data interpretation should be undertaken such that the results are directly applicable to the proposed activity.
- chemical concentrations can be compared to the PSQGs. These have been developed using a large amount of data based on biological effects on benthic organisms. Thus, comparison with the various effect levels will provide a reasonably accurate indication of potential environmental effects.
  - at present there are no guidelines for biological analyses (i.e., tissue residues).
  - the proponent should ensure that the required statistical analyses are also performed. This is especially important for dredging assessments.
  - the proponent should check the requirements in the guidelines to be used to ensure that all criteria are met through the sampling program.
- 3) Compare patterns of contamination among different parameters. In many areas where point sources of contaminants are a concern, more than one contaminant is often involved. Data interpretation should seek to establish where and to what degree relationships between different contaminants exist.

- e.g., correlation statistics among different parameters. These can relate the occurrence of one contaminant with that of another, and may be useful where more than one contaminant is or has been released from a source.
  - correlation of chemical contamination with physical characteristics of the area. Can test whether chemical parameters are related to physical parameters such as grain size. Chemical parameters can also be tested in relation to biological measures such as numbers of taxa, density of specific taxa, etc.
  - measures such as the Percentage of Similarity of Community developed by Johnson & McNeill (1986) can be used to test differences in benthic communities among stations or over time at the same stations.
- 4) Establish linkages between sediment chemical concentrations and sources. This will require familiarity with sources and the detailed industrial processes, as well as the behaviour of the contaminant in the receiving water.
- direct cause-effect relationships between sediment contamination and sources can be difficult to establish. Presence of compound in sediments in discharge area and absence or significantly lower concentrations outside is usually best link that can be achieved.
  - requires data on sources, processes used in the sources, fate and effects of the compounds.
  - may require use of statistical tests to help establish the validity of these relationships.
- 5) Establish linkages between chemical concentrations and biological effects. The analysis should seek to determine causal relationships between contaminant concentrations and:
- community level effects
  - availability and uptake
  - toxicity of the compound at the concentration present through assessment of chronic and

acute effects (e.g., laboratory sediment bioassay)

Data interpretation in support of these would normally involve statistical analyses.

## SECTION VIII REFERENCES

- Baudo, R. 1990. Ch. 2. Sediment Sampling, Mapping, and Data Analysis. *In*: Baudo, R, J. Geisy and H. Muntau. *Sediments: Chemistry and Toxicity of In-Place Pollutants*. Lewis Publishers, Chelsea, Michigan. 405 pp.
- Baxter, M.S., J.G. Farmer, I.G. McKierley, D.S. Swan and W. Jack. 1981. Evidence of the unsuitability of gravity coring for collecting sediment in pollution and sedimentation in rate studies. *Environ. Sci. Technol.* 15, 843-846.
- Cummins, K.W. 1975. Chapter 8: Macroinvertebrates. *In*: Whitton, B.A. [ed] *River Ecology*. Univ. California Press, 1975. 725 pp.
- Cuff, W. and N. Coleman. 1979. Optimal Survey Design: Lessons from a Stratified Random Sample of Macrobenthos. *J. Fish Res. Bd. Canada.* 36: 351-361.
- Elliott, J.M. 1977. Some Methods for the Statistical Analysis of Samples of Benthic Invertebrates. 2nd. Ed. *Freshwat. Biol. Assoc. Publ. No. 25.* 156 pp.
- Green, R. H. 1979. *Sampling Design and Statistical Methods for Environmental Biologists*. Wiley, New York, N.Y.
- Hynes, H.B.N. 1960. *The Biology of Polluted Waters*. Univ. Toronto Press, Toronto. 202 pp.
- Hynes, H.B.N. 1970. *The Ecology of Running Waters*. Univ. Toronto Press, Toronto. 555 pp.
- Johnson, M.G. and O.C. McNeil. 1986. Changes in Abundance and Species Composition in Benthic Macroinvertebrate Communities of the Bay of Quinte, 1966-84. *In*: Minns, C.K., D.A. Hurley

- and K.H. Nicholls [eds.] Project Quinte: Point-Source Phosphorus Control and Ecosystem Response in the Bay of Quinte, Lake Ontario. *Canad. Spec. Publ. Fish. Aquat. Sci.* 86.
- Karr, J.R. 1987. *Biological Monitoring and Environmental Assessment: A Conceptual Framework*. *Environ. Management* 11: 249-256.
- Legendre, L. and P. Legendre. 1983. *Numerical Ecology*. Elsevier Sci. Publ. Co. 1983.
- Mudroch, A. and S.D. MacKnight. 1991. *CRC Handbook of Techniques for Aquatic Sediments Sampling*. CRC Press. Boca Raton, Florida. 210 pp.
- OMOE (Ontario Ministry of Environment). 1978. *Water Management, Goals, Policies, Objectives and Implementation Procedures of the Ministry of Environment*. Ontario Ministry of Environment, Toronto, Ontario, pp. 70.
- OMOE (Ontario Ministry of Environment). 1983. *Handbook of analytical methods for environmental samples*. Ontario Ministry of Environment, Toronto, Ontario.
- Persaud, D., R. Jaagumagi and A. Hayton. 1992. *Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario*. OMOE Toronto. 30pp.
- Persaud, D. and W.D. Wilkins. 1976. *Evaluating Construction Activities Impacting on Water Resources*. Ontario Ministry of the Environment.
- Resh, V. H. 1979. *Sampling Variability and Life History Features: Basic Considerations in the Design of Aquatic Insect Studies*. *J. Fish. Res. Bd. Canada*. 36: 290-311.
- Sly, P.G. 1969. *Bottom Sediment Sampling*. *Proceed. 12th Conf. Great Lakes Research*. 883-898.
- Thomas, R.L. and A. Mudroch. 1979. *Small Craft Harbours Sediment Survey - Lakes Ontario, Erie and St. Clair*. Report to Small Craft Harbours Branch - Ontario Region.

## Summary

### Sediment Sampling Planning Guide

#### 1. Identify Objectives

These could include any or all of:

- establish baseline data
- monitor impacts of discharge
- research on fate and effects
- establish suitability of dredged material
- state of the environment reporting

#### 2. Planning

Desk-Top Activities

- assemble all historical and current information
- using suitable maps identify:
  - water uses
  - access points
  - extent of study area
  - sources (discharge points)
  - plot information from historical/preliminary studies
- determine sampling density in consideration of:
  - statistical requirements
  - sources of contaminants
  - extent of contamination
  - historical information
- determine study components using:
  - known sources or extent of contamination
  - chemical behaviour of contaminants, including expected pathways of exposure or uptake by organisms
  - regulatory requirements (e.g., PSQGs)

Field Activities

- reconnaissance survey
  - verify historical information
  - verify sources/discharges
  - determine equipment necessary
- select appropriate sampling device
- sampling schedule including contingency plans

#### 3. Sample Collection

- collect into appropriate containers
- collect required quantities
- ensure required protocols are followed based on requirements of analytical labs.

#### 4. Data Interpretation

- apply tests and analyses appropriate to the study objectives
- determine need for statistical analysis

## Appendix 1

### Sources of Historic Information

All sediment studies require some preliminary information upon which a study plan is founded. This is especially important for studies related to dredging and to possible sediment remediation.

All provincial Lakefill projects are required to submit an Environmental Assessment and the sampling program outlined here is intended to address these concerns as well. Proponents are advised to review the requirements for EAs to determine what information will be required.

The proponent should review all existing data for the proposed lakefilling, dredging or remediation area. All potential data sources should be checked such as:

- **Site-Specific Data:** This includes data most relevant to the project review. Data collection may have been undertaken by OMOE, Environment Canada, a proponent such as Canada Department of Public Works (PWC) or a university or other research organisation. The data may be available only in the files of the regulatory agencies, of proponents (PWC) or of funding agencies (e.g., Harbour Commissions, Transport Canada). Some data may have been published in the form of data or technical reports published by government departments, e.g., Thomas and Mudroch (1979), Persaud *et al.* (1985), or as theses or research publications from universities.
- **Area or Regional data:** This includes data collected on a regional basis and may include the area under consideration, but is not specific to it. Such data may be used to predict ranges of concentrations to be expected as a minimum in the area, but it must be remembered that certain areas are typically contaminant "sinks" and will generally be found to have higher concentrations. An example would be Thomas and Mudroch (1979), where the chapters describing each lake clearly show the differences between the lakes. Typically, area or regional data have been collected by such groups as universities, OMOE, Environment Canada or Fisheries and Oceans Canada as part of a general description of the environment of the Great Lakes. Since each agency will have undertaken studies for different purposes it should not be assumed that all relevant parameters have been measured.
- **Point-Sources and Outfall data:** This would include monitoring data for various effluents into a harbour. Data may be available from OMOE or Environment Canada, or possibly a municipality. Data should be reviewed in the context of identifying a potential problem, e.g., elevated concentrations of chromium in the area of a metal plating industry.
- **Other Data Sources:** This includes data from a variety of sources where the data can be used to predict or indicate the nature of the material. For example, geotechnical borehole logs may indicate layers of black organic-rich silt on a sand layer; the silt layer may be contaminated. Such information may be useful in the prediction of problems or hot-spots. The data will generally be in the files of agencies responsible for harbour construction projects.



## Appendix 2

### OPERATIONAL EVALUATION OF SAMPLING DEVICES (from Sly, 1969)

---

<u>GRAB SAMPLERS</u>	<u>Characteristics</u>
Franklin-Anderson Grab	Suitable for obtaining material for bulk sample analysis. Works best in soft clays, muds, silts and sands. Will occasionally obtain a good gravel sample. Material of no use for structural or other specific analyses.
Dietz-LaFond Grab	Can be used for general sampling but not recommended for any particular use. Of all the samplers tested, this pattern proved to be the least suitable.
Birge-Ekman Dredge	Suitable for soft clays, muds, silts and silty sands. This sampler should be used under calm water conditions, typically in small lakes or restricted areas. The lack of sample disturbance, square cross section and moderate penetration make this sampler suitable for detailed studies (i.e., biological and geochemical) of the top 2 to 3 cm of bottom sediment. Because of its light weight and easy handling, it is well suited to small boat operations.
Petersen Grab	This sampler, like the Franklin-Anderson, is suitable for taking bulk sample material in most types of sediment. It is quite unsuited for studies of detailed and specific sediment properties, though it is perhaps a little more successful in taking gravel samples. Either of these two samplers (Petersen or Franklin- Anderson) will do well as a general purpose bulk sampler.
Ponar Grab	An excellent general purpose bottom sampler. In practice it operates better than either the Petersen or Franklin-Anderson over the full range of bottom types. It can also obtain samples with little or no disturbance and with the protecting screens removed or folded back, direct access can be had to the sediment surface of the sample. Such access to undisturbed samples makes it suitable for geochemical, sedimentological, biological and structural studies. Because of the large sample volume and its relatively undisturbed state, this sampler is very suitable for population studies of the bottom sediment fauna.
Shipek Grab	An excellent general purpose sampler, though perhaps a little heavy for small boat operation. This sampler is capable of working with almost equal success on all types of bottom materials. It provides a sample even less disturbed than the Ponar, making it the most suitable sampler (under test) for detailed geological studies of the sediment surface. The sampler volume is significantly less than that of the Ponar, and the quantity of material sampled at maximum cutting depth is also less than the Ponar. These two points may, therefore, favour the Ponar for certain biological (population) studies. On the other hand, the rapid rotation of the Shipek bucket, as opposed to the much slower closure of the Ponar's jaws, may make it more suitable for sampling sediment containing a significant population of non-sessile forms.

### Grab Trigger System Reliability

Franklin Anderson Grab	Good, but perhaps too sensitive on hard sand and gravel bottom.
Dietz-LaFond Grab	Poor, unless area of trigger foot is increased to at least 50 cm <sup>2</sup> . Triggering may often be impossible in very soft mud unless the foot has been modified.
Birge-Ekman Dredge	Good. Triggered by messenger weight dropped from surface, normally consistent but can be affected on soft bottoms if sampler is allowed to settle for too long before dropping the messenger.
Petersen Grab	Fair to good, though tends to be a little over-sensitive on hard sand and gravel bottoms.
Ponar Grab	Good, though like the Petersen, tends to be a little over-sensitive on gravel bottoms.
Shipek Grab	Good, though some slight settlement may occur before triggering on very soft materials. Sampler may fail to trigger when lowered gently on soft bottoms. By lifting and dropping the trigger weight a few centimetres after bottom contact, abortive casts may be avoided. The slight movement of the inertial trigger weight has no other affect on the sampler.

### Grab Jaw Shape, Design and Cut

Franklin-Anderson Grab	Poor. During the first stages of closure and when under the greatest pressure of springs and weight, the jaw shape loosely follows the arc of cut. However, the degree of fit becomes progressively worse as the closing pressure is reduced. Because each jaw is semi-cylindrical in shape, sample displacement is necessary within it if anything near maximum capacity is to be achieved.
Dietz-LaFond Grab	Poor. As for Franklin-Anderson.
Birge-Ekman Dredge	Excellent. Jaw shape exactly follows arc of cut and almost no sample displacement occurs.
Petersen Grab	Poor. Comments as for Franklin-Anderson, except that instead of the reduction in closure pressure being produced by slackening of tensional springs, the same result is effected by reduced leverage on the scissor arms mounted across the hinge line.
Ponar Grab	Excellent. Jaw shape exactly follows arc of cut and almost no sample displacement occurs.
Shipek Grab	Excellent. As for Ponar. In addition, the rotation of the bucket is extremely rapid. In most cases, the rotational shear is far greater than the sediment shear strength, thus the cutting action is very clean (producing minimal

disturbance), particularly in soft clays, muds, silts and sands.

### Preservation and Protection from Washout in Grabs

Franklin-Anderson Grab	Fair, but the tightness of closure is largely dependent upon the lack of grains trapped between the edges of the jaws. Providing tight fit between the two jaws is obtained, the sample is well shielded against washout. If the jaws are kept open by material trapped between the jaws, washout can be severe or total.
Dietz-LaFond Grab	Fair. Comments as for Franklin-Anderson.
Birge-Ekman Dredge	Good, except when the sampler is used in very coarse or shelly sediment. Under these conditions, material may be trapped between the jaws, preventing their closure. In this case, washout may be severe. The jaws are so designed that they slightly overlap one another, thus a slight imperfection of closure can be tolerated.
Petersen Grab	Good. Comments as for Birge-Ekman.
Ponar Grab	Good. Comments as for Birge-Ekman. In addition to the overlap jaws, this sampler has a pair of metal side plates, mounted close to the moving side faces of the jaws. These plates further reduce the possibility of washout.
Shipek Grab	Excellent. The great advantage of the Shipek, over all of the other samples described, is that the bucket closes with its separation plane aligned in the horizontal rather than in the vertical. Good samples can be retrieved even when bucket closure is prevented by pebbles or similar material, even 2 to 5 cm across. With the bucket properly rotated, washout is completely avoided.

### Stability

Franklin-Anderson Grab	Fair. Despite the weight of this grab, it tends to "stream" at an inclined angle under conditions of rapid ship drift or fast water flow. Provided lowering conditions are calm and stable, the sampler will hold upright during the initial sampling process; if, however, the line is allowed to slack, the sampler will fall over.
Dietz-LaFond Grab	Poor. This sampler is very sensitive to "streaming" and will rarely operate in the vertical position unless used in ideal conditions. Its tendency to maintain an inclined attitude during descent sometimes results in a failure to trigger.
Birge-Ekman Dredge	Fair. Despite the light weight of this sampler and its tendency to "stream", its wide base gives good stability and stance once it has come to rest on the sediment floor. Under poor sampling conditions, however, it becomes impossible to operate because: (a) the sampler, due to its light weight, is continually being lifted and dropped and "streamed" along the bottom and (b) any slack in the line, particularly near the sampler, is likely to impede the

proper function of the triggers' messenger weight. It tends to roll over after triggering on all but soft bottoms.

Petersen Grab

Good. This is a heavy sampler with a wide base line (when the jaws are open). It maintains a near vertical descent under all conditions, but after sampling it tends to fall over (unless on a soft bottom).

Ponar Grab

Very good. Comments as for Petersen; because of its weight and wide baseline (when jaws are open), this grab has a good vertical descent under most conditions and has a stable stance on the bottom. The presence of the fixed side plates prevents the grab from falling over after jaw closure and helps in preserving a near perfect bottom sample.

Shipek Grab

Excellent. Despite the large size of this Grab sampler, its weight ensures a near perfect vertical descent even under conditions of rapid drift or fast water flow. The sampler is also very stable even on bottom slopes 20 degrees or more. This stability ensures the minimum possible disturbance of the sample material.

## CORE SAMPLERS

### Characteristics

Benthos Gravity Corer

Cores of 3 m or less in soft clays, muds or sandy silts, particularly suitable for studies of the sediment/water interface, for studies on depositional sediment structures.

Alpine Gravity Corer

Cores of 2 m or less in almost all sediment types. The rugged nature of his corer lends itself to general usage. For studies involving sediment structure or large volumes of material, the corer is unsuitable; for studies of a pilot nature, or to prove the suitability of an area for piston coring, this gravity corer is excellent.

Phleger Corer

Cores of 0.5 m or less, in almost all sediment types, particularly suited to bottom materials containing a high percentage of fibrous organic material. The low cutter angle, the narrow wall thickness and high point loading, and the extremely sharp cutter, make it very suitable for sampling shallow lacustrine and estuarine deposits, marsh deposits and thin peat beds.

Multiple Corers

Still under investigation but early results indicate are very effective in soft sediments where a number of replicates are required or spatial heterogeneity is high.



